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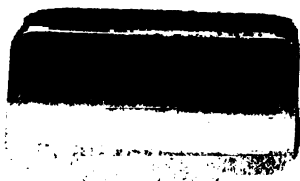
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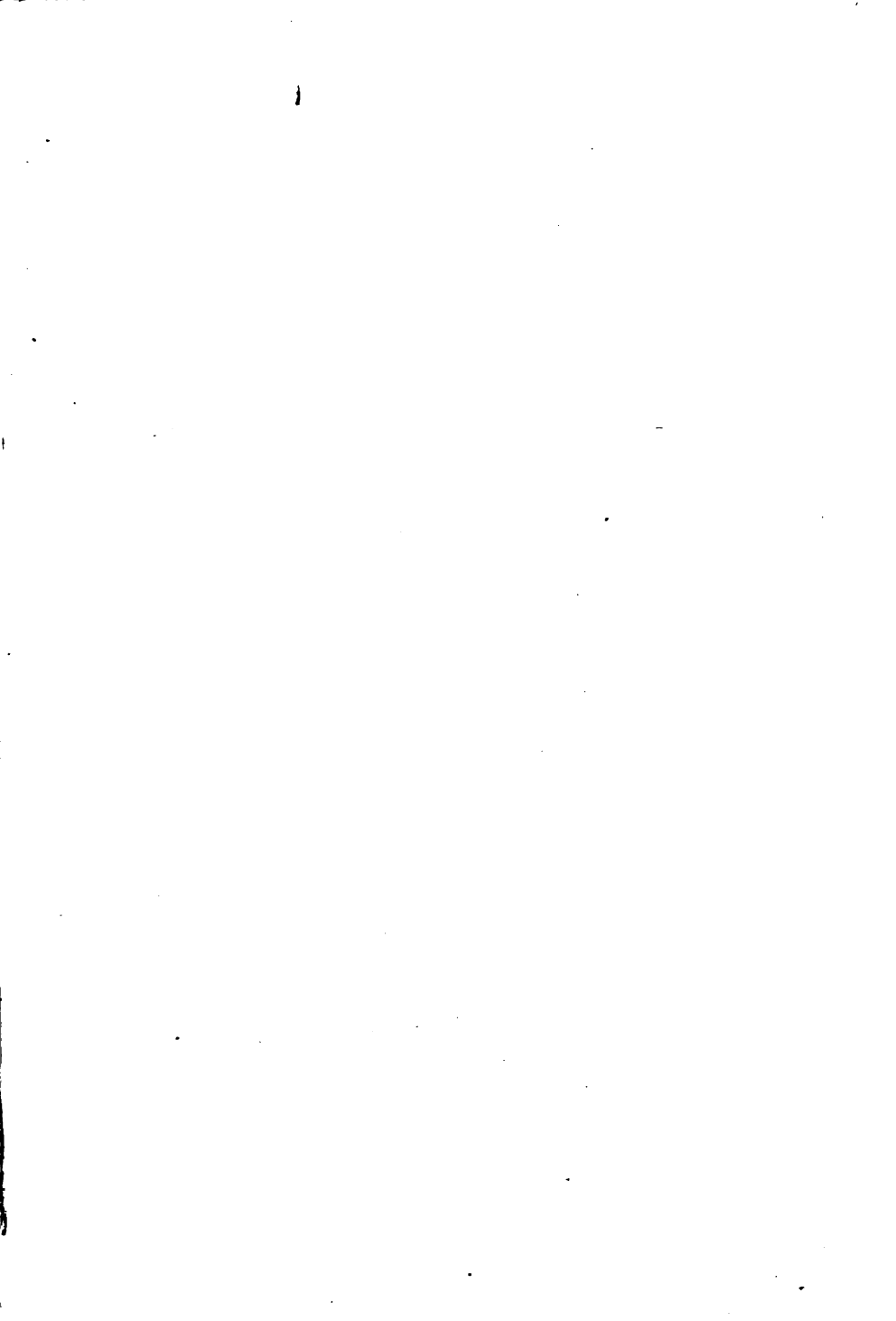
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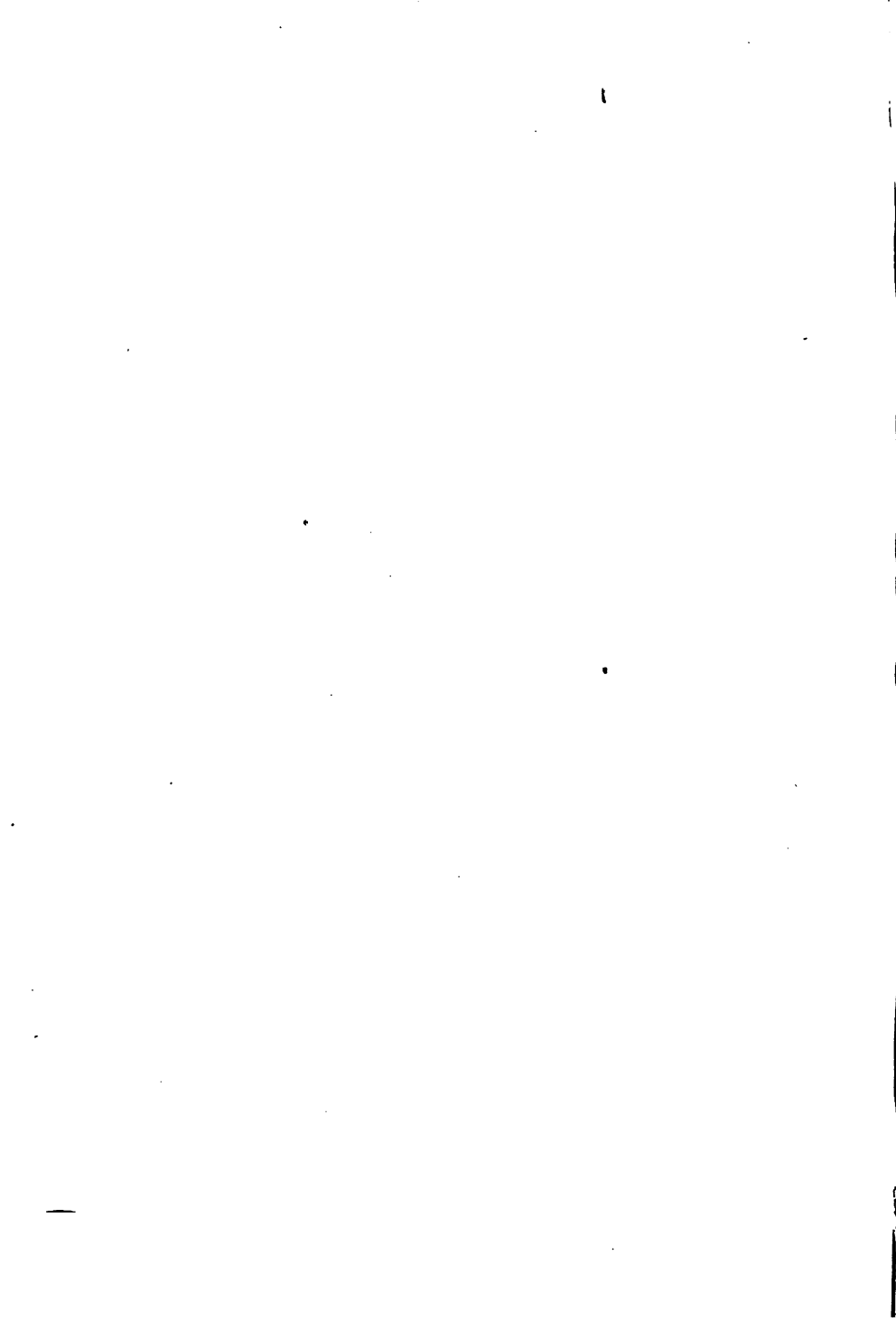
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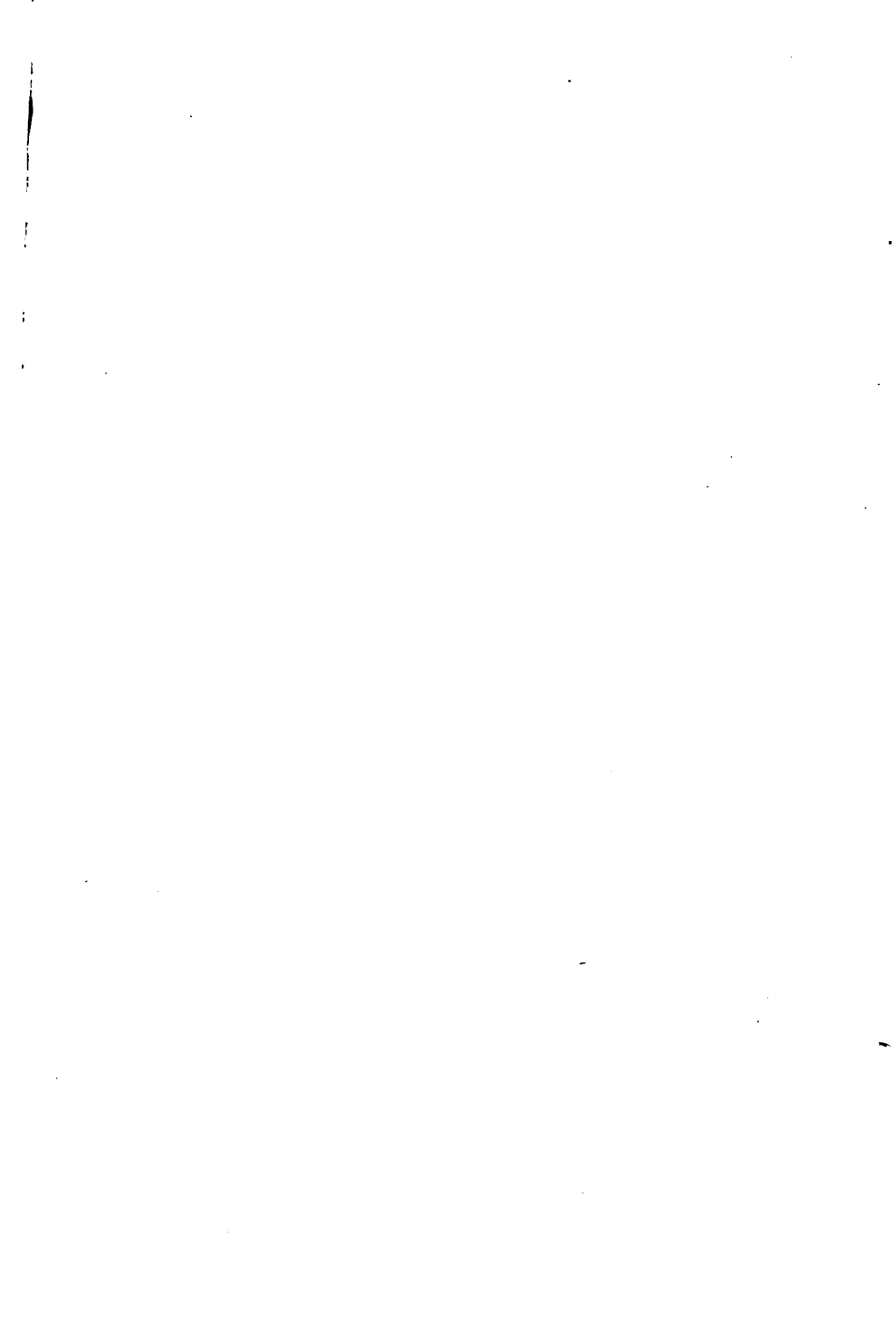
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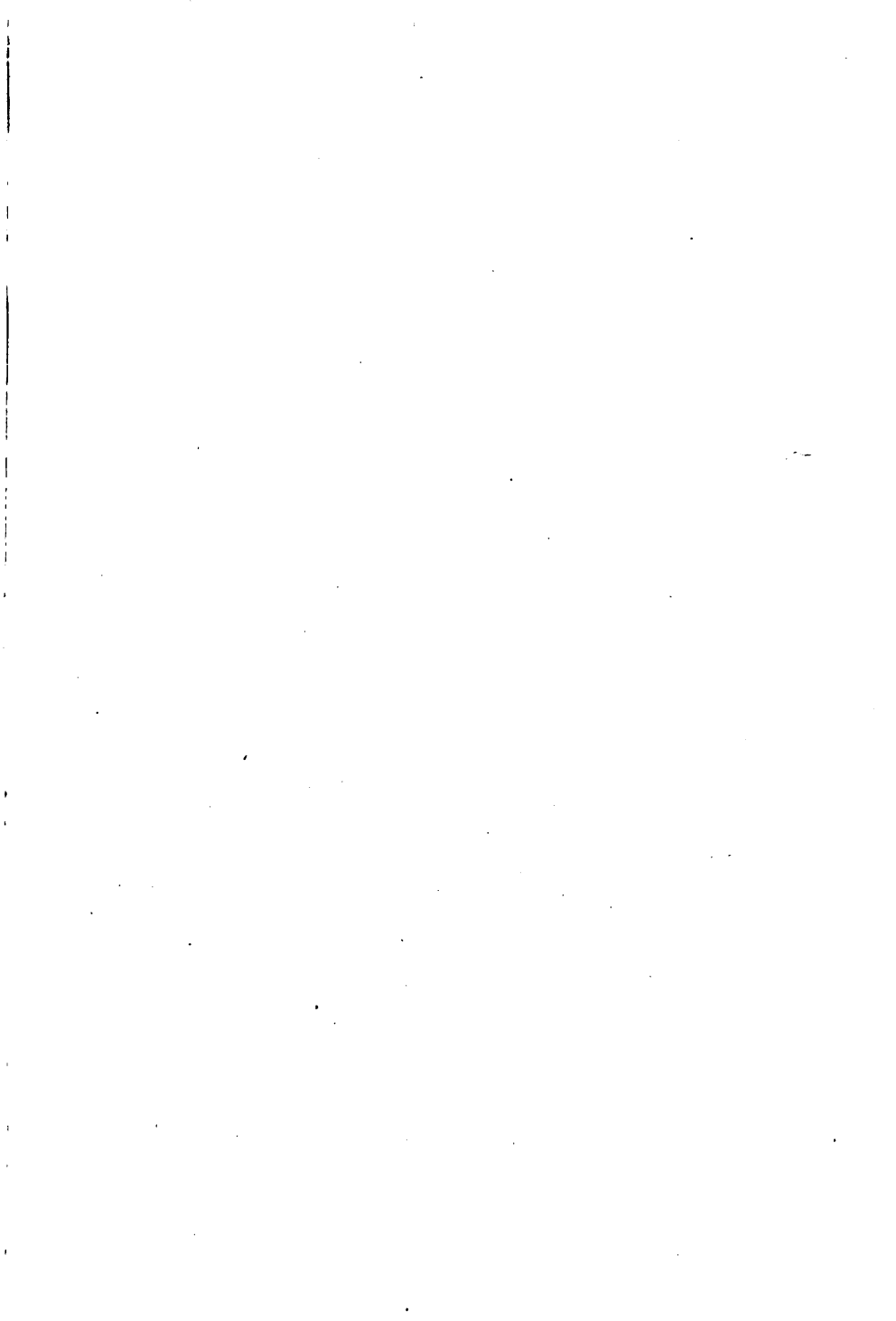
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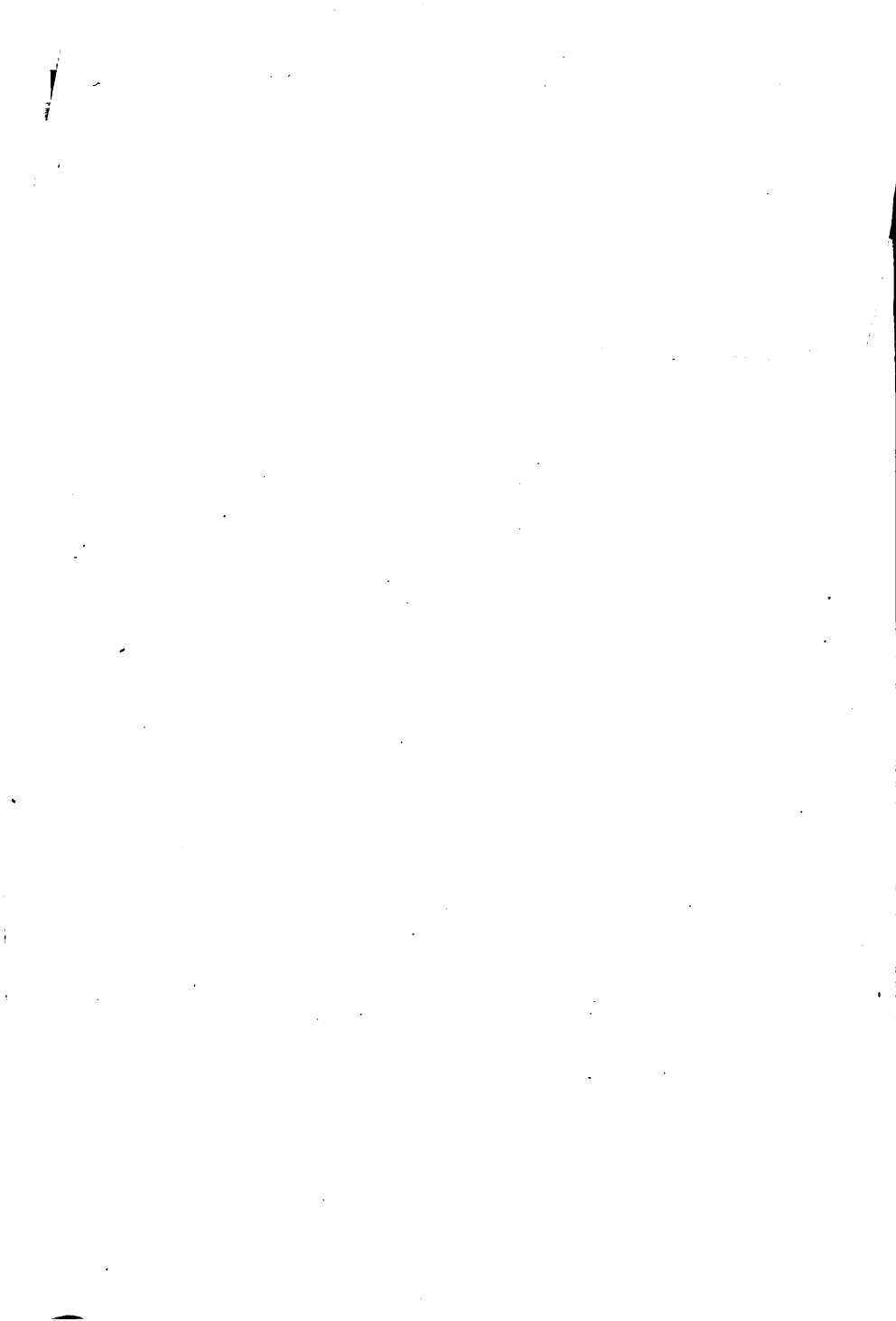












An Introduction to the Methods of Analysis
IN USE IN THE
Laboratory of Sanitary Chemistry of Cornell University

PART I.
THE ANALYSIS OF WATER
FOR
Household and Municipal Purposes

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TAYLOR & CARPENTER
Ithaca, N. Y.
1911

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PREFACE.

This little book, the first of a series dealing in an elementary manner with the methods for the examination of water and food, has been prepared in order to place in the hands of our students directions which are believed to present in a more satisfactory manner the special practices and apparatus in use in the Chemical Laboratories of Cornell University, than can be done by the use of existing manuals, and at the same time provide more thorough training in manipulation, in the preparation of reagents and in the standardization of solutions for volumetric analysis.

The subject matter is devoted to laboratory methods mainly, and is intended to supplement the lecture courses on Sanitary Chemistry in which the interpretation of results, relative value of different methods, the taking, transportation and preparation of samples is discussed at length.

In the main, the official methods of the Laboratory Section of the American Public Health Association have been followed, but the authors have not hesitated to modify these methods where, in their judgment and experience such changes seemed desirable either from a pedagogical or practical viewpoint.

E. M. C.

H. W. R.

INTRODUCTION.

The student of Sanitary Chemistry should ever bear in mind that the Water Analyst almost invariably has a complex problem to solve in the case of every sample of water submitted to him for examination. He is called upon to answer the following questions: 1. Is the water in question potable? 2. If not, what is the trouble? 3. Is it possible to form an opinion as to the future quality of the water? 4. Can the water be improved in quality, and how? 5. Does the water contain constituents rendering it unsatisfactory or undesirable for general household or municipal use? 6. Is the water suitable for general industrial purposes?

It is thus essential that the analyst shall have clearly before him these questions that he may so obtain his samples and so plan his analyses as to enable him to form an opinion and properly advise his client.

The complete data necessary for the formation of a reliable opinion as to the fitness or desirability of a water for a municipal or household water supply and to properly answer the above questions may be grouped under the following heads.

I. HISTORY OF THE SAMPLE—its source; the topography and geology of the surrounding country.

This we may term environmental data.

II. PHYSICAL EXAMINATION.

Temperature

Appearance

Turbidity (sediment and suspended matter)

Color

Odor

Taste

III. CHEMICAL EXAMINATION.

Determinations of:—

1. Nitrogen content in ammoniacal salts or compounds, in "albuminoid" or protein-like material, and in the form of oxidized nitrogen, or in other words:

Nitrogen as "Free Ammonia" (or Saline Ammonia)

Nitrogen as "Albuminoid Ammonia"

Nitrogen as Nitrites

Nitrogen as Nitrates

Nitrogen as "Total Organic Nitrogen"

2. "Organic Matter" as shown by the quantity of oxygen required to oxidize the material in solution; this value is termed "oxygen consumed" and is expressed in parts per million of oxygen used. It generally conforms closely to the carbon content of the organic matter present.

3. Chlorine in the form of chlorides.

4. Alkalinity expressed in terms of calcium carbonate.

5. Mineral Acid Hardness—expressed in terms of calcium carbonate. This value has been variously termed "Permanent Hardness," "Normal Hardness," "Incrustants," etc.

6. Total Hardness expressed as calcium carbonate.

7. Total Solid Residue left on evaporation and drying at 110°C.

8. Solids in suspension.

9. Loss of Solids on ignition, also designated as "Organic and Volatile Matter."

10. Phosphates.

11. Sulphates.

12. Iron and Manganese.

13. Free Carbon Dioxide and Carbon Dioxide as Bicarbonates and as Normal Carbonates.

14. Oxygen in solution; usually designated as "Dissolved Oxygen."

15. Soap consuming power—calculated from the Alkalinity and Mineral Acid Hardness or actually measured by means of a soap solution.

In addition to the above determinations there may be required:

A. The analysis of the non-volatile solid residue for the purpose of learning (1) the character of the scale which will be formed in steam generators, or (2) the presence of substances causing corrosion of boiler tubes, or (3) causing foaming or priming in the boiler.

B. Search for poisonous inorganic or organic compounds.

C. Special studies of the water with reference to unusual or very abnormal conditions or with reference to certain industries.

IV. BACTERIOLOGICAL EXAMINATION

Comprising:—

1. Estimation of the number of bacteria capable of growing at low temperatures (18° to $20^{\circ}\text{C}.$) on standard culture media in air.

2. Estimation of the number of bacteria capable of growing at blood heat (38° to $40^{\circ}\text{C}.$) on standard media in air.

3. Search for the presence, character and approximate number of bacteria capable of producing gas (fermenting) in dextrose, lactose, and also in certain specific cases, sucrose.

4. Search for the presence of organisms capable of forming hydrogen sulphide from albuminoids, a group of organisms, which, for want of a better term at the present time, may be designated as "Putrefactive Organisms."

5. Search for the presence and probable number of bacteria whose normal habitat is believed to be the intestinal

tract of man and animals. Usually designated on a report as "Fecal and Intestinal Organisms," or "Fecal and Sewage Organisms."

6. Search for the presence of certain definite pathogenic organisms suspected of being present, as, for example, the organism associated with typhoid fever, or cholera, or anthrax, etc., etc.

7. Search for the presence of anaerobic organisms, (Data of much value in many instances).

8. Search for the presence of certain organisms which, if found, would throw light upon the character of the water or upon the presence or absence of certain kinds of polluting or contaminating material.

V. MICROSCOPICAL EXAMINATION.

Comprising:—a microscopical study of the sediment and suspended matter including:—

1. Qualitative and quantitative studies of the plankton of the water.

2. Special investigations as to the cause of disagreeable odors and tastes.

3. Special investigations of suspended matter in the study of sources of pollution, etc.

It is, of course, evident at once that every sample of water submitted for examination will not require being subjected to each one of the individual tests or determinations enumerated above. The proper choice of methods of procedure must depend upon the judgment of the analyst and the problem involved. But it should be emphatically stated that it is rare indeed that an analyst does too much, and it is generally the case that a water examination is so incompletely carried out, that the results reported are insufficient to permit the formation of dependable answers to the questions at issue.

WORK PRELIMINARY TO THE CHEMICAL EXAMINATION OF A SAMPLE OF WATER.

Before any analyses of water samples may be undertaken it is essential that all the necessary apparatus shall be placed in a useable condition, all volumetric solutions shall be standardized and all reagents and culture media shall be prepared and adjusted.

In order that this work may be expeditiously done, suggestions will be found on page 106, Appendix A, relative to the order in which to proceed so as to make the best use of the time at the student's disposal while following the course in Water Analysis as given by the authors, and outlined in this book.

Solutions and Reagents to be Prepared.

STANDARD SOLUTIONS OF CAREFULLY ADJUSTED VALUES.

Hydrochloric Acid Tenth Normal.

Sodium Hydroxide Tenth Normal.

Soda Reagent Tenth Normal.

Sulphuric Acid Tenth Normal.

Sulphuric Acid Fiftieth Normal.

Potassium Permanganate. 1 cc. to be exactly equivalent to 0.0001 gm. available oxygen.

Oxalic Acid. 1 cc. to be exactly equivalent to 1 cc. of the above permanganate solution.

Sodium Chloride. 1 cc. to be exactly equivalent to 0.001 gm. of chlorine.

Silver Nitrate. 1 cc. to be exactly equivalent to 0.001 gm. chlorine.

Potassium Nitrate. 1 cc. to contain exactly 0.0001 gm. Nitrogen as Nitrate.

Potassium or Sodium Nitrite. 1 cc. to contain exactly 0.000001 gm. Nitrogen as Nitrite.

Iron Solution. 1 cc. to contain exactly 0.0001 gm. of Iron.

Sodium Phosphate. 1 cc. to contain exactly 0.0001 gm. PO_4 .

Potassium Dichromate. For determination of SO_4 . 1 cc. to be exactly equivalent to 0.001 gm. SO_4 .

Potassium Dichromate. For standardizing thiosulphate. 1 cc. to be exactly equivalent to 0.01 gm. Iodine.

Potassium Permanganate. 1 cc. to contain exactly 0.0001 gm. Manganese.

Sodium Thiosulphate. Tenth Normal.

Potassium Acid Sulphate (Primary Potassium Sulphate). Fiftieth Normal.

Sodium Arsenite, Tenth Normal.

Iodine, Tenth Normal.

SPECIAL REAGENTS.

Phenol Disulphonic Acid Reagent for Nitrates.

Ilosvay-Lunge Reagent for Nitrites.

Nessler Reagent for Ammonia.

Alkaline Potassium Permanganate Reagent.

Ethyl Orange—Indicator (or Methyl Orange).

Phenolphthalein, Indicator.

Erythrosin—Indicator.

Chloroform—Neutral for use with Erythrosin.

Potassium Chromate—free from chlorides—Indicator for Determination of Chlorine.

Ammonium Molybdate Solution.

Potassium Sulphocyanate Solution.

Sodium Carbonate Solution, saturated with the fused salt.

Barium Chromate (solid).

Potassium Hydroxide for Oxygen Dissolved.

Sulphuric Acid for Oxygen Dissolved.

Sulphuric Acid 1:4.

Potassium Hydroxide for Nitrates.

Nitric Acid for Manganese.

Dilute Nitric Acid for Manganese.

Sulphuric Acid for Manganese.

Iodide-Starch Paper.

PREPARATION OF SOLUTIONS.

STANDARD HYDROCHLORIC ACID, N/10.

Prepare 1 liter in the usual manner, by obtaining the specific gravity of the concentrated HCl. Determine its strength by consulting the table, page 111 giving the weights HCl per 100 cc. of acids of different densities. Make the solution slightly too strong (Why?) using freshly boiled distilled water (Why?). Employ a one liter graduated cylinder rather than a measuring flask (Why?).

Now determine the relation existing between this approximately N/10 acid and the approximately N/10 sodium hydroxide solution prepared as on page 11, but not yet standardized, using 10 cc. of acid, 100 cc. of boiled distilled water and 1 cc. of the adjusted phenolphthalein indicator. This relationship must be determined with great care. (What is gained by this titration?)

Determine the absolute strength of the approximately N/10 HCl by precipitating with silver nitrate, collecting the silver chloride formed on Caldwell-Gooch filters, and drying to constant weight at 140°C .

In the preparation of the Gooch crucibles be sure that the porcelain crucible and the platinum disk are scrupulously clean, and that the suction pump at your work table is in proper order and yields a strong and steady exhaust. The asbestos pulp poured over the disks should be of such

a thickness that the positions of the tiny perforations may be just discerned. When all the possible moisture has been removed by the pump, dry over a Bunsen lamp with a very low flame (Why?), when thoroughly dry, ignite, cool in a desiccator and weigh accurately to tenths of a milligram.

From a burette, deliver into a 250 cc. glass stoppered Erlenmeyer flask, 25 cc. of the standard HCl, add 100 cc. of distilled water, and 5 cc. of concentrated HNO_3 (Why?), now add 5 cc. more than the volume of silver nitrate solution (obtained from the stock room, strength 42 gms. per liter), required to completely precipitate the HCl. (Why calculate?). Insert the stopper in the flask, cover with a towel or cloth (Why?) and shake violently (Why?). As soon as the precipitate has settled, decant the supernatant liquid through the previously weighed Caldwell-Gooch filter, being very careful to transfer as little of the silver chloride as possible on the filter (Why?). Wash the precipitate with repeated very small (Why?) volumes of 1 per cent. HNO_3 (Why?), each time carefully decanting through the Gooch filter. After three or four washings with from 50 to 75 cc. of dilute HNO_3 , test a portion of the filtrate to ascertain whether the washing has been complete (How?). Finally transfer all of the precipitated AgCl to the crucible, dry at 140° for one hour, cool in a desiccator, weigh and replace in the air bath again for a short time and again weigh (Why?). Measure out a second portion of the HCl solution and precipitate with silver nitrate in exactly the same manner.

The two parallel determinations must differ from each other not more than 0.5 milligram. The entire process of standardization should be completed in about two hours.

From the average of the two weights of silver chloride, determine the exact strength of the standard acid. From this value calculate the volume of exactly N/10 acid which

would have been required to neutralize the amount of approximately N/10 sodium hydroxide solution used in the preliminary titration.

Now calculate the volume of distilled water which must be added to the acid in the measuring cylinder in order that it shall be made *exactly* N/10; add this volume of water less about 0.5 cc. (Why?).

Having thus obtained an acid of supposedly *exactly* N/10 strength, again titrate a known volume with the approximately N/10 sodium hydroxide solution using phenolphthalein as indicator. This volume should correspond exactly with the calculated volume obtained as previously described.

STANDARD SULPHURIC ACID, N/10. 1 LITER.

Determine the specific gravity of the concentrated acid and ascertain its strength in SO_3 from the table, page 112. Calculate the volume required to prepare one liter of N/10 acid. Make the solution in boiled distilled water slightly too strong (Why?). Titrate with the standardized N/10 NaOH, using phenolphthalein as indicator. Dilute to exact strength.

STANDARD SULPHURIC ACID, N/50. 1 LITER.

Prepare 1 liter of standard acid of this strength by diluting the exactly N/10 H_2SO_4 with slightly less than the required volume of boiled distilled water. Check the correctness of the acid by titrating against the N/10 NaOH. This solution of H_2SO_4 must be adjusted so as to be exactly N/50. Calculate and record in your note book the value of 1 cc. of this N/50 acid in terms of Ca; CaO ; CaCO_3 ; $\text{Ca}(\text{HCO}_3)_2$; $\text{Ca}(\text{OH})_2$; Mg; MgO ; MgCO_3 ; $\text{Mg}(\text{HCO}_3)_2$; Na_2CO_3 ; NaHCO_3 .

STANDARD SODIUM HYDROXIDE SOLUTION, N/10. 1 LITER.

Weigh out a few grams more than twice the weight of

NaOH required to make a N/10 solution. Place in a 500 cc. flask, pour in 20 to 25 cc. of freshly boiled distilled water and stir by rotating until the surfaces of all the pieces of sodium hydroxide have become thoroughly washed off. Pour off and reject this solution (Why?). Dissolve the remaining hydroxide in freshly boiled water, keeping the flask stoppered. Titrate an aliquot part as described under N/10 HCl on page 9.

From the final titrations of this solution made in the process of standardizing the N/10 HCl solution, calculate the volume of distilled water required to reduce the strength to exactly N/10. It is always wiser to add about 1 cc. of water less than the calculated value (Why?) and then titrate again with the exactly adjusted HCl.

The determination and adjustment of the strength of both the N/10 HCl and N/10 NaOH must be done with great care and accuracy since these two solutions serve to adjust the strength of all other alkalies and acids employed in the analysis of water samples.

Calculate and record in your note book the value of this solution in terms of Cl (HCl); SO_4 (H_2SO_4); and H ions.

STANDARD "SODA REAGENT," N/10. 1 LITER.

This standard solution has been thus designated by the Committee on Standard Methods of Water Analysis of the American Public Health Association. It consists of a solution containing equal weights of NaOH and Na_2CO_3 so diluted as to be *exactly* tenth normal in strength.

It is made with washed NaOH and dry Na_2CO_3 dissolved in boiled water, using a little less than the calculated volume of water. Titrate an aliquot part with N/10 sulphuric acid, using erythrosin as an indicator. (Why not use phenolphthalein?)

(Would a mixture of equal volumes of N/10 NaOH and

N/10 Na_2CO_3 yield a solution conforming to the definition of the Soda Reagent?)

STANDARD SODIUM CHLORIDE. 1 LITER.

1 cc. of this solution to be equivalent to exactly 0.001 gram of combined chlorine.

In a clean mortar grind a portion of the absolutely pure salt. Weigh out on a watch glass more than the required amount of the salt and dry at 110°C . to constant weight. From this dried portion weigh out very carefully but rapidly the exact quantity required to yield a solution of the desired strength, transfer to a liter measuring flask and fill to the mark.

If no pure sodium chloride is available a solution may be prepared from the commercial salt, somewhat too strong. To determine the strength, aliquot portions are taken, acidulated with nitric acid, precipitated with silver nitrate and the resulting silver chloride weighed on a Caldwell-Gooch filter, the process being substantially the same as that described for the standardization of HCl. From the value thus obtained dilute to the exact strength required.

STANDARD SILVER NITRATE. 500 CC.

1 cc. to be equivalent to exactly 0.001 gm. of chlorine as chloride.

A few days before this solution is to be prepared, fill a one liter bottle with distilled water, add a crystal of silver nitrate about the size of a grain of wheat and when dissolved allow the solution to stand in the light for a few days in direct sunlight if possible. (What is thus accomplished?) Filter (Why?) and add to 500 cc. of the filtrate a trifle more than the weight of silver nitrate required for a solution of the strength indicated above.

Determine the exact strength of this silver solution as follows:—From a burette run into a No. V evaporator, ex-

actly 10 cc. of the standard sodium chloride solution (1 cc. equals 0.001 gm. Cl) already prepared, add 90 cc. of *filtered* distilled water, then 1 cc. of potassium chromate indicator (see page 27) using a 1 cc. pipette and stir well. Run into this solution from a 10 cc. burette graduated in 1/20 cc. the standard silver solution whose strength is to be determined, being careful to run in the silver solution very slowly and to stir continually. The titration should be conducted under yellow light or observed through goggles provided with yellow glasses (Why?). The end point is reached upon the appearance of the first pink or brownish tint as seen by yellow light. A recognition of the end point is facilitated, if a second evaporator containing 100 cc. of distilled water and 1 cc. of chromate indicator is placed, during the process, alongside of that containing the solution to be titrated. Make two or three titrations so as to be sure of the results obtained. Calculate the amount of dilution required to bring the silver solution to the proper strength. Adjust the strength, using some of the water treated with silver nitrate and sunlight, but being careful to add less than the calculation calls for, then titrate again.

Silver solutions used as in this method with chromate as indicator should never be made by dissolving the exact quantity of silver nitrate required by theory, nor may the solution be standardized by precipitating the silver as chloride and weighing.¹

Instead of water first treated with silver nitrate, re-distilled water obtained by the process mentioned under the permanganate solutions may be employed to even better advantage. Such standard solutions are more permanent than those prepared as described above.

¹ Hazen, American Chemical Journal, 11, 409 (1889).

STANDARD POTASSIUM PERMANGANATE SOLUTIONS.

A. For Determination of "Oxygen Consumed." 1 liter.

This solution must be of such strength that 1 cc. shall yield exactly 0.0001 gm. of oxidizing oxygen when used with H_2SO_4 in the presence of organic matter. Calculate the weight of the salt required for one liter of solution. Make up the solution very slightly too strong, shake until all of the permanganate has dissolved and allow the solution to stand for two or three days before adjusting the strength (Why?); then filter if necessary, and determine the strength of the resulting solution by means of two or three separately, very carefully weighed out portions of ferrous ammonium sulphate. (What weight of ferrous ammonium sulphate will be required to reduce 10 cc. of the permanganate solution?)

Introduce the weighed out portion of ferrous ammonium sulphate into a 200 cc. Erlenmeyer flask, add 10 cc. of 1:4 H_2SO_4 and 50 to 100 cc. of distilled water (Why is it best to add the acid first?). Run in the permanganate until the faintest tint of pink remains permanent for several minutes. From the titration values obtained calculate the volume of water required to bring the permanganate solution to the strength required. If the standard solution shows an appreciable deposit on standing the three days, there should be added to adjust the strength a little less than the calculated volume of water (Why?); this difference generally amounts to about 1 cc. in 50 cc. of ordinary distilled water.

Potassium permanganate solutions of the above strength are not permanent when made up with ordinary distilled water, but it has been shown¹ that if the water used has been redistilled from permanganate and sulphuric acid, a very permanent solution is obtained.

¹ Chamot. The Determination of Oxygen Consumed in Water. Amer. Jour. Pub. Hyg., 18, 280 (1908).

B. For Determination of Manganese. 1 liter.

This solution shall contain in 1 cc. 0.0001 gm. Mn. Calculate the weight required and standardize the solution with ferrous ammonium sulphate as directed above. Finally adjust the strength by the proper amount of dilution.

STANDARD OXALIC ACID SOLUTION. 1 LITER.

For Determination of "Oxygen Consumed."

This solution shall be of such strength as to exactly reduce the standard permanganate solution (A) volume for volume. Calculate the weight of oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) required. Make the solution a little stronger than necessary, using filtered, freshly boiled distilled water and rinsing the measuring cylinder or flask with boiling water to kill moulds or fungi. After standardizing and adjusting the strength of the permanganate solution, determine the strength of the oxalic acid as follows.

Measure from a burette into a 400 cc. Erlenmeyer flask, 10 cc. of the oxalic acid, add about 190 cc. of distilled water, 10 cc. of sulphuric acid (1:4), and heat almost to boiling. Run in from a burette the standard permanganate until a permanent pink color just results. From the titration values thus obtained dilute to the proper strength and titrate again.

STANDARD SODIUM CARBONATE, N/22. 500 cc.

Prepare 250 cc. of an approximately N/10 solution of Na_2CO_3 , using distilled water which has been redistilled from barium hydroxide, the distillate being collected in a receiver having an atmosphere free from CO_2 (How may this be accomplished?).

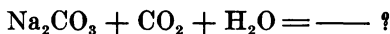
A less satisfactory method (Why?) for preparing the water consists in thoroughly boiling it in a flask and allowing it to cool in an atmosphere free from CO_2 .

Determine the strength of the sodium carbonate solu-

tion thus prepared, by titrating an aliquot part (after diluting with boiled distilled water) with N/10 hydrochloric acid using ethyl orange as indicator in one portion and erythrosin as indicator in another portion. (Is it possible to use phenolphthalein? Explain fully.)

From the value obtained calculate the volume of the carbonate solution and the volume of CO_2 free water necessary to prepare 500 cc. of exactly N/22 Sodium Carbonate Solution.

The standard solution is used for the determination of "Free Carbon Dioxide." The reaction being



Find the value of 1 cc. of this standard solution in terms of CO_2 .

STANDARD NITRITE SOLUTION, 500 CC.

A solution of such strength that 1 cc. shall contain 0.001 milligram of Nitrogen as NO_2 .

It is customary to prepare this solution from silver nitrite, but experiments have shown that the pure potassium or sodium nitrites of commerce are sufficiently pure to permit of their being employed as standards. Powder some potassium nitrite (whose purity has been tested) and dry it in a desiccator over calcium chloride to constant weight. Weigh out accurately such a weight of the dry salt that if dissolved in 100 cc. of distilled water free from nitrites and 10 cc. of this solution be then diluted to one liter, 1 cc. of the weaker solution will have the required strength (1 cc. = 0.001 mg.) in Nitrogen as Nitrite.

(Or, if very accurate results are required: Prepare silver nitrite. Recrystallize several times from hot water, dry to constant weight. Weigh out the proper amount to give a solution containing from 100 to 1000 times the required amount of nitrogen as nitrite. Dissolve in water, precipitate with sodium chloride and filter from the silver

chloride formed. Dilute to known volume in a measuring flask. Measure out a portion of this stock solution and dilute in a measuring flask with nitrite free water to the proper strength. This solution is not permanent.)

FUCHSINE STANDARDS FOR DETERMINATION OF NITRITES.¹

Prepare a stock solution by dissolving about 0.1 gram Fuchsin in 1 liter boiled water. Standardize this solution or dilutions thereof with a standard nitrite solution. By proper dilutions prepare a series of color standards for use in the colorimeter, e.g. solutions corresponding in value to 0.001; 0.005; 0.010; and 0.015 parts per million nitrogen as nitrite.

STANDARD NITRATE SOLUTION. 500 cc.

Powder and dry to constant weight at 100°C. potassium nitrate known to be absolutely pure. Weigh out an amount of this salt such that when dissolved in 500 cc. of water, 1 cc. will contain exactly 0.0001 gm. of Nitrogen as Nitrate.

STANDARD IODINE SOLUTION, N/10, 500 cc.

Dissolve 9 grams pure white potassium iodide in 500 cc. water. Transfer to a bottle and add a trifle more than the calculated weight of iodine required for a tenth normal solution. Shake at intervals until all the iodine has dissolved.

Weigh out very exactly into a 250 cc. Erlenmeyer flask such a weight of pure barium thiosulphate as will require about 10 cc. of the iodine solution for its oxidation to tetrathionate. Add 100 cc. of distilled water, a few drops of soluble starch indicator and run in from a burette the iodine solution until a blue color appears. From the value found adjust the iodine solution so that it is exactly tenth normal.

Instead of starch indicator methylene blue may be

¹ Amer. J. Pub. Hyg., 20, 363 (1910).

used and the iodine solution added until the blue color is discharged.

(Write all reactions involved.)

STANDARD POTASSIUM DICHROMATE.

(A) *For Colorimetric Determination of SO_4 . 500 cc.*

Read method used in the determination (page 41) writing the equation for the reactions involved and then calculate the weight of potassium dichromate required to make 500 cc. of a solution 1 cc. of which will be exactly equivalent to 0.001 gm. SO_4 .

Very carefully weigh out the calculated weight of the C. P. recrystallized salt, transfer to a 500 cc. measuring flask, dissolve, fill to the mark, mix thoroughly and transfer to a bottle for use.

(B) *For Standardizing Thiosulphate Solutions. 500 cc.*

Read the directions given on page 19 for standardizing the sodium thiosulphate. Write the equation for the reaction,—



Then calculate the weight of dichromate required to make 500 cc. of a solution of such strength that 1 cc. will liberate 0.01 gram of iodine from potassium iodide. Weigh out this amount very exactly, dissolve in a 500 cc. measuring flask and fill to the mark.

STANDARD SODIUM THIOSULPHATE SOLUTION. 1 LITER.

Tenth Normal with reference to iodine.



Having calculated the weight required, weigh out roughly from one-quarter to one-third more (Why?) than this amount. Dissolve in one liter of distilled water and filter if necessary.

To determine the strength of the thiosulphate solution proceed as follows. Into a 500 cc. glass stoppered Erlen-

meyer flask measure from a burette 20 cc. of standard potassium dichromate solution (see page 19, 1 cc. dichromate equivalent to 0.01 gm. I.) and from a pipette 10 cc. of potassium iodide solution (page 26), mix and add 100 to 150 cc. of distilled water. Mix thoroughly. Now add 5 cc. of concentrated hydrochloric acid. Insert the stopper and shake. After standing for about five minutes, dilute to about 300 cc. and run in, from a burette, the thiosulphate solution whose strength is to be determined, until the dark color changes to a straw yellow. Now add 1 cc. of soluble starch indicator and continue the titration until the blue color just disappears, or add 1 cc. of dilute methylene blue indicator and continue until the blue color of the indicator appears. With properly prepared indicators the end point in either case is very sharp.

The number of cubic centimeters of thiosulphate used multiplied by . . . (?) is equivalent to 1 gm. of iodine?

STANDARD SODIUM ARSENITE, N/10, 500 cc.

This solution is made tenth-normal with reference to iodine and is employed for the determination of the "available chlorine" in calcium hypochlorite ("bleach").

Dissolve about 12 grams of pure primary sodium carbonate in about 200 cc. of distilled water, heat to boiling and add slowly a little more than the proper weight (calculated) of finely powdered pure arsenic trioxide, heat until all has dissolved, cool, transfer to a 500 cc. measuring cylinder and fill to the mark.

Determine the strength of the sodium arsenite solution thus prepared by measuring out from a burette 10 cc. into a 250 cc. Erlenmeyer flask, dilute, add a few drops of soluble starch indicator and run in from a burette standardized N/10 iodine solution until a blue color appears, calculate and add the volume of water necessary to make the arsenic solution exactly N/10.

(Write equations for all reactions, basing them upon $\text{As}_2\text{O}_3 + \text{I} + \text{Na}_2\text{O} = ?$)

STANDARD IRON SOLUTION.¹ 500 cc.

1 cc. to contain exactly 0.0001 gm. Fe.

Weigh out very carefully the calculated amount of non-effloresced crystals of ferrous ammonium sulphate, add 10 cc. of sulphuric acid (1:4) and 50 cc. of distilled water, warm gently and add very carefully dilute permanganate solution until the iron is just oxidized but no more. Dilute to 500 cc. in a measuring flask.

STANDARD PHOSPHATE SOLUTION. 500 cc.

1 cc. to contain 0.0001 gm. PO_4 .

Weigh out very exactly the calculated weight of pure freshly recrystallized secondary sodium phosphate, $\text{Na}_2\text{HPO}_4 \cdot 12 \text{H}_2\text{O}$. Dissolve in a measuring flask and fill to the mark.

Since water acts upon glass, dissolving silica, very accurate determinations are not possible unless the standard is kept in wax or rubber bottles. (Why?) See Determination of PO_4 in Water on page 40).

STANDARD POTASSIUM ACID SULPHATE, N/50. 500 cc.

Dissolve the crystallized salt in 500 cc. of freshly distilled water. Titrate against carefully adjusted N/50 ~~sulphuric acid~~ *NaOH* using ethyl or methyl orange as indicator. Check the results of this standardization by titrating against your N/22 sodium carbonate solution using phenolphthalein as indicator.

(Write all the reactions involved.)

STANDARD AMMONIUM CHLORIDE SOLUTION.

1 cc. to contain exactly 0.00001 gm. of Nitrogen as NH_3 .

¹ Leffmann, Examination of Water.

Pure ammonium chloride is finely powdered and dried in a water oven to constant weight. Such an amount is accurately weighed out and dissolved in ammonia free water in a 100 cc. measuring flask and filled to the mark, with ammonia free water, so that if 10 cc. be taken and diluted to one liter one cubic centimeter of the weaker solution will contain 0.00001 gm. nitrogen.

Note. This solution will be found prepared and ready for use in the laboratory.

AMMONIA FREE WATER.

The authors prefer the following method for its preparation. A five liter balloon flask is three-fourths filled with distilled water, from 5 to 10 cc. of sulphuric acid (1:4) are added and sufficient potassium permanganate added to give a distinct pink color. The water thus treated is distilled through a Hempel tube, the first 100 cc. rejected, the next portion of distillate tested and if ammonia free the distillate is collected until all but about 200 to 500 cc. have passed over.

Not infrequently the laboratory distilled water may be boiled in an open balloon flask and after about one quarter has been evaporated the remainder will be found ammonia free. There is, however, sufficient organic matter and nitrites present so that the product obtained does not long remain ammonia free.

Note. Ammonia Free Water will be found in a tubulated bottle ready for use.

NESSLER REAGENT, 250 CC.

Dissolve about 13 grams of potassium iodide in 25 cc. of distilled water, add with constant stirring a cold saturated solution of mercuric chloride until the precipitate at first formed no longer redissolves, being careful to pour no crystals of mercuric chloride into the iodide solution and to

avoid the formation of more than just a very little permanent precipitate. Filter the solution of the double iodide of potassium and mercury. Dissolve ~~35~~ 35 grams of potassium hydroxide in 150 cc. of water, allow to stand in a corked flask until clear. Decant the clear solution from the deposit and add it to the double iodide solution. Dilute the mixture to 250 cc. Mix well and add drop by drop with constant stirring a saturated solution of mercuric chloride until a slight yellowish permanent precipitate results. Allow to stand until clear, and decant into a capped bottle for use. 55

This reagent should have a distinct yellow color, should give a brown tint within one minute when 0.5 cc. is added to 10 cc. of distilled water and must not give a turbidity when 0.5 cc. is added to 8 cc. of standard ammonium chloride solution diluted to 10 cc. with ammonia free water in a Nessler tube.

REAGENTS FOR NITRITES.¹

1. Weigh out approximately 0.100 gm. of pure (white) Alpha-Naphthylamine. Boil gently IN THE HOOD in a covered beaker with about 30 cc. of distilled water until the compound is almost completely dissolved, adding water to replace that lost by evaporation. Filter while hot through a small wet filter IN THE HOOD to remove any insoluble coloring matter. Add at once to the filtrate 150 cc. of 30 per cent. acetic acid.

2. Dissolve 0.500 gm. of pure sulphanilic acid in 150 cc. of 30 per cent acetic acid. Filter.

3. Mix the two solutions and keep in several small tightly stoppered bottles. The reagent thus prepared should be practically colorless. If pink, nitrites were present in the distilled water or have been absorbed from the air during

¹ Ilosvay-Lunge. Zeit. angew Chem., 2, 666 (1889); Lunge, Tech. Methods Chem. Anal. (Lunge-Keane) I, pt. 2, 760.

the process of preparation. The addition of a very tiny quantity of nitrite free zinc dust will decolorize (Why?) the reagent and render it fit for use.

When many Bunsen burners are in use in the laboratory and hood it is next to impossible to obtain a colorless reagent (Why?).

ALKALINE PERMANGANATE REAGENT.

Dissolve 50 grams of potassium hydroxide in 125 cc. of distilled water.

Dissolve 2 grams of potassium permanganate in 125 cc. of distilled water.

Pour the alkali slowly, with constant stirring, into the permanganate. Mix well; transfer to a porcelain evaporator and boil gently until about one-third of the volume has been driven off (Why?). When cool, dilute to 250 cc. with ammonia free water. Keep in a bottle tightly stoppered with a rubber stopper.

POTASSIUM HYDROXIDE SOLUTION FOR OXYGEN DISSOLVED.

Prepare 100 cc. of potassium hydroxide solution of such strength that 1 cc. will precipitate approximately one and one-half times as much ferrous iron as is contained in one cc. of a ferrous ammonium sulphate solution of which 1 cc. requires .0003 gram of oxygen for oxidation. This potassium hydroxide solution need not be standardized (Why?) and may be kept as a stock solution.

SULPHURIC ACID SOLUTION FOR OXYGEN DISSOLVED.

Prepare 100 cc. of sulphuric acid of such strength that 1 cc. will neutralize 2 cc. of the above alkali.

This need not be standardized (Why?) and may also be kept in stock.

SULPHURIC ACID; 1:4. 500 cc.

Add 100 cc. of concentrated C. P. sulphuric acid to

400 cc. of distilled water. Add slowly with constant stirring. Never mix the liquids in a bottle.

SULPHURIC ACID FOR MANGANESE DETERMINATION. 500 cc.

Into 500 cc. of distilled water, pour cautiously with constant stirring 13 cc. of concentrated C. P. sulphuric acid. Add slowly drop by drop potassium permanganate solution until a very faint permanent pink color results.

NITRIC ACIDS. FOR MANGANESE DETERMINATION.

A. Dilute. 25 cc. of concentrated HNO_3 . 475 cc. of water.

B. Sp. Gr. 1.135. 25 cc. of concentrated HNO_3 . 75 cc. of water.

SATURATED SODIUM CARBONATE SOLUTION.

Make 20 cc. of saturated sodium carbonate (Na_2CO_3). Allow the solution to stand until all of the undissolved salt has settled. Decant the clear solution.

BARIUM CHROMATE. 1 GRAM.

Used in the dry state in colorimetric determinations of SO_4 . The salt is prepared by adding to a hot solution of barium chloride sufficient hot potassium chromate solution to give a permanent yellow color to the liquid above the precipitated barium chromate. Wash the precipitate by decantation with distilled water acidified with acetic acid, until the wash water is colorless in a 100 cc. Nessler tube. Then transfer to the filter and wash with distilled water until practically neutral. Dry and rub to a fine powder. From one to one and one-half grams of the dry salt will be needed.

Calculate the weight of barium chloride required, and the weight of chromate necessary to completely precipitate the barium, allowing for losses due to solubility, etc.

Note. Barium chromate will be found on the reagent shelves.

AMMONIUM MOLYBDATE SOLUTION. 200 cc.

Dissolve 10 gms. of pure ammonium molybdate in freshly drawn (Why?) distilled water. Dilute to 200 cc. This solution is employed in the colorimetric determination of PO_4 and soon becomes unfit for use if kept in glass bottles (Why?).

POTASSIUM IODIDE SOLUTION. 100 cc.

Dissolve 15 grams of pure potassium iodide in 100 cc. filtered distilled water. This solution is used in connection with standard dichromate for determining the strength of thiosulphate solutions.

SILVER SULPHATE SOLUTION.

Prepare a solution of such strength that 1 cc. will be equivalent to 0.001 gram of chlorine. This reagent is employed in the determination of nitrogen as nitrate.

PHENOL DISULPHONIC ACID REAGENT FOR NITRATES.¹

Dissolve 25 grams of pure white phenol in 150 cc. of concentrated sulphuric acid, add 75 cc. of fuming sulphuric acid (13% SO_3), stir well and heat for two hours in an air bath at about 100°C .

POTASSIUM HYDROXIDE SOLUTION FOR NITRATE DETERMINATION. 50 cc.

This potassium hydroxide solution should be of such strength that 10 cc. will neutralize 4 cc. of the phenol disulphonic acid. (What will be the strength of such a solution in grams per liter, assuming that 2 cc. of the phenol disulphonic acid will be equivalent to 4 cc. of concentrated sulphuric acid?)

POTASSIUM SULPHOCYANATE SOLUTION. 500 cc.

Dissolve 25 grams of potassium sulphocyanate in 500 cc. of water. Filter if necessary.

¹ See Papers by Chamot, Pratt and Redfield. J. Am. Chem. Soc., 31, 922 (1909); 32, 630 (1910); 33, March 1911.

POTASSIUM CHROMATE SOLUTION.

Dissolve 10 grams of the pure salt in 100 cc. of distilled water. Add, drop by drop, a solution of silver nitrate (Why?) until a permanent red precipitate results. Filter. Dilute the clear filtrate to 200 cc.

CHLOROFORM (NEUTRAL).

This reagent is used with the erythrosin indicator. Commercial chloroform or the pure product kept in clear glass bottles almost invariably contains acid compounds which would vitiate all results obtained in determinations of alkalinity. These acid reacting substances may be removed by shaking the chloroform in a separatory funnel with sodium carbonate solution, drawing off and shaking with distilled water at least six times to remove any of the alkaline carbonate. Test with a portion of the adjusted indicator to prove that it is neutral and preserve in black glass containers.

(Neutral chloroform ready for use will be found on the reagent shelves.)

INDICATORS.

Note. All indicators will be found on the reagent shelves. Each student should, however, carefully adjust about 25 cc. of the stock solutions for personal use.

Caution. In standardizing alkalies or acids for water analysis it is always best to dilute, with boiled distilled water, the volumes taken for titration to the same volume as that of the water samples which will be used for analysis and to employ in every titration carefully measured volumes of the indicator. In other words, always have exactly similar conditions in titrations for standardizing as in titration of water samples.

Phenolphthalein.

Dissolve 1.0 gram in 200 cc. of 50 per cent. alcohol.

Filter. Add *dilute* potassium hydroxide very cautiously drop by drop until the faintest discernible permanent change of color results. Use boiled water in making the dilute alcohol and boil the 50 per cent. alcohol in a flask before using.

Erythrosin. (Sodium salt of Tetra-iodo-fluorescein).

Dissolve 0.1 gram in one liter of distilled water. Titrations are best made by shaking in a stoppered bottle. For 100 cc. of water use 2 cc. of erythrosin and 5 cc. of neutral chloroform. (See Determination of Alkalinity, page 45).

Ethyl Orange (Sodium salt of Para-diethyl-amino-azobenzene Sulphonic Acid.)

The authors prefer this compound to methyl orange since it gives sharper end points, although not so sensitive as the methyl compound there is less difficulty in recognizing the change from alkaline to neutral.

For use in water analysis prepare a saturated aqueous solution. Carefully adjust its reaction by the addition of a few drops of dilute hydrochloric acid and allow to stand for a few days. Filter for use, and dilute with 90 parts water.

Para Nitro Phenol.

Dissolve 1. gm. in 200 cc. of 50 per cent. alcohol. Filter. The solution should be almost colorless. If deep yellow add acid; if colorless, add alkali until the faintest possible yellow tint appears. This indicator may be employed in place of ethyl or methyl orange.

Lacmoid (Resorcin Blue).

Dissolve 0.2 gm. of the commercial product in 100 cc. of 95 per cent. alcohol, filter. Adjust reaction if necessary.

This indicator behaves somewhat like litmus and hence in alkalinity determinations, boiling is necessary (see page 47). The common mineral acid salts of iron, aluminum, copper and zinc are practically neutral to lacmoid, but acid

to litmus. Free mineral acids in solutions of these salts may therefore be determined.

Potassium Iodide Starch Paper.

This indicator paper may be obtained at the stock room ready for use.

It may be prepared as follows. Rub up about 3 grams of potato or arrowroot starch in an evaporator with 250 cc. water. Heat to boiling with constant stirring until all the starch granules are destroyed, then add 1 gram potassium iodide and 1 gram crystallized sodium carbonate. Dilute to 500 cc., saturate strips of filter paper with the mixture, dry them and preserve in a stoppered bottle for use.

“CLEANING MIXTURE.”

Pour into a No. 8 *Evaporator*, 150 cc. of distilled water, add 40 gms. of commercial potassium dichromate and warm gently until all the salt is dissolved. Cool the solution and add *very slowly with constant stirring* 230 cc. of concentrated commercial sulphuric acid. Allow the thick mixture to cool, and then transfer to a 500 cc. wide mouthed, glass stoppered bottle.

This chromic-sulphuric acid mixture is used for cleaning burettes, pipettes and certain bacteriological apparatus. It may be used repeatedly, as long as crystals of chromic acid remain in the mixture. Shake before using.

The mixture is powerfully corrosive and great care must be exercised to avoid dropping it on the work-table or floor. In the event of any being spilled, dilute at once with much water and wipe up with a sponge.

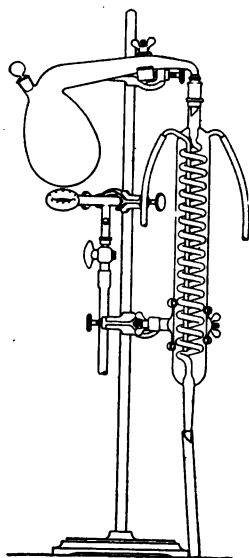
METHODS OF ANALYSIS.

DETERMINATION OF FREE AND ALBUMINOID AMMONIAS.

Preliminary Operations.

Clean very thoroughly the set of Nessler tubes and the retort and condenser using chromic acid cleaning mixture if necessary. Rinse thoroughly with tap water or finally with a very little ammonia free water. Tap water is preferable to distilled water for the final rinsing since it contains only traces of ammonia. The apparatus being properly prepared clamp the spiral condenser in a vertical position at such a height as to permit the placing of a 10 cc. Nessler tube easily under the condenser, yet having the end of the condenser extending just into the tube. Place a stout rubber band around the upper tube of the condenser and push it down as far as it will go. Place a similar band around the end of the retort, clamp the retort in place with its end entering the condenser tube as far as it will go. The retort is to be held by the clamp alone and must not be supported by a ring or wire gauze, since the heating is to be done over the free flame of a small ring burner. A tight joint is made between retort and condenser by winding a piece of tin foil, $2\frac{1}{2}$ to 3 cm. wide by 6 or 7 cm. long around the two tubes and holding this seal in place by the rubber bands, see Fig. 1. Two condensers and retorts are thus set up, one for free ammonia, the other for albuminoid ammonia. The apparatus thus set up must next be freed from all ammonia as follows.

A. *For Free Ammonia.* Introduce into the retort 50 cc. of distilled water and 2 cc. of a saturated solution of sodium carbonate. When waters very high in calcium or



-Fig. 1-

magnesium salts are to be analyzed a few tiny pieces of pumice stone should also be introduced (Why?). Distil off 30 cc., then 10 cc. into a clean Nessler tube. Turn off the gas and as soon as the 10 cc. distillate has reached room temperature add 0.5 cc. of Nessler reagent. If at the end of five minutes no yellow color is seen in the tube, looking down through it when held over white paper, the apparatus is ammonia free, and ready for the sample. If a yellow color appears introduce into the retort as soon as it is cool 40 cc. of distilled water, distil 30 cc., reject, then collect the next 10 cc. and test as above.

B. For Albuminoid (Total) Ammonia. Introduce into the retort 10 cc. of alkaline permanganate reagent and 50 cc. of distilled water. Pumice stone is to be used under the same conditions as provided for under free ammonia. Distil off 40 cc., reject, then collect the next 10 cc. in a Nessler tube. Nesslerize as stated above. If after five minutes no yellow color appears the apparatus is ammonia free and ready for the sample. If not found to be ammonia free, add 40 cc. of distilled water, distil 30 cc., reject, then collect the next 10 cc. and test, and repeat if necessary.

Process.

I. Free Ammonia. Introduce into the retort, which has been purified as directed above, 100 cc. of the sample. Distil, collecting the distillate in 10 cc. Nessler tubes and regulating the rate of heating so that it requires at least

two minutes to fill a tube to the mark. After five fractions have been obtained, stand the sixth in warm water (not hot) and add Nessler reagent. If no yellow color results, shut off the gas. If ammonia is still coming over in the sixth tube distil over a total of eight tubes and discontinue the heating.

After the distillates have acquired room temperature add in turn to each 10 cc., 0.5 cc. of Nessler reagent always beginning with the last tubes. Should a deep color appear in the third distillate, it is probable that the quantity of ammonia in tubes one and two will be too great to allow them to be treated; in such an event pour the contents of tube No. 2 into a 50 cc. measuring flask free from ammonia, dilute to the mark with ammonia free water, mix, pour 10 cc. into a Nessler tube, add the reagent and allow to stand. Should the color still be too dark, a portion of the dilute sample is again diluted with a known volume of ammonia free water. Distillate No. 1 is to be treated in like manner. The quantity of ammonia in each of the fractions is determined as described below.

II. *Total Ammonia and Albuminoid Ammonia.* Introduce into the retort, which contains the alkaline permanganate, and which has been first purified as directed above (B), 100 cc. of the sample. Distil, collecting the distillate in 10 cc. Nessler tubes at the rate of two minutes for each 10 cc. Test for complete expulsion of ammonia just as described under I. and proceed as there directed.

Determination of the Quantity of Ammonia Present in the Distillates.

Prepare a series of color standards as follows: Introduce into a series of 10 cc. Nessler tubes, the following volumes of standard ammonium chloride (1 cc. = ~~X~~0.00001 gm. N) 0.5 cc.; 1 cc.; 2 cc.; 3 cc.; 4 cc.; 5 cc.; fill each tube to the mark with ammonia free water, stir thoroughly with

a glass plunger (washing the plunger with tap water after each tube is mixed) and add to each tube 0.5 cc. of Nessler reagent. After standing five minutes compare the colors of the distillates from the water sample with the standards, holding the tubes side by side over white paper and looking down through them. To make sure of the matching of the tints, place the tube containing the sample first on one side then on the other of the standard (Why?). The intensity of the color being proportional to the amount of ammonia present, it of course follows that tubes having the same intensity must contain equal amounts of ammonia.

From the amount of nitrogen as total ammonia found by distilling with alkaline permanganate, subtract that obtained by distillation with sodium carbonate (nitrogen as free ammonia), the difference being called nitrogen as albuminoid ammonia.

The authors believe that the obtaining of the albuminoid nitrogen in this manner is preferable to the direct determination as usually practiced, namely adding the alkaline permanganate to the residue after the free ammonia has been expelled, the chief objections to this latter procedure are, (1) the impossibility of properly controlling the concentration, sometimes all the free ammonia is expelled in the first three tubes while at other times six, seven or even eight tubes must be obtained; (2) it is impracticable to wholly free the alkaline permanganate from ammonia, hence a correction must always be applied; (3) most polluted waters contain nitrogenous products which distil over with the free ammonia but which do not give a color with Nessler, but these volatile compounds are almost invariably so acted upon by alkaline permanganate as to yield ammonia. Circumstances not infrequently arise when a determination of the nitrogen as total ammonia is inadvisable or impracticable. In such an event proceed as follows:

First determine the amount of ammonia contained in the alkaline permanganate. This is accomplished by first distilling ammonia free water in the retort until the distillate running from the condenser gives no color with Nessler reagent. Cool, introduce 10 cc. of alkaline permanganate and sufficient ammonia free water to make about 100 cc. Distil until a fraction is obtained, giving no color with Nessler. Determine the total quantity of N as ammonia in the distillates as usual.

Determine the N as free ammonia in the usual manner, then add to the water residue in the retort 10 cc. of alkaline permanganate and a volume of ammonia free water equal to about one-half or two-thirds of the volume of the free ammonia distillates. Distil, collecting the distillates in 10 cc. Nessler tubes as usual. Determine the N as ammonia present in these distillates and from the sum subtract the equivalent quantity of N as ammonia found in the alkaline permanganate. The difference gives the N as albuminoid ammonia.

When dealing with waters containing H_2S or sulphides, it has been the practice of the authors for many years to add lead acetate, filter through an ammonia free filter, and distil. Bartow and Harrison¹ have recently suggested adding sulphuric acid to the sample, boiling until all H_2S is expelled and then proceeding as usual.

When the amount of free ammonia in a water is very high it may be determined by adding to the sample in a 50 cc. or 100 cc. Nessler tube the proper volume of Nessler solution, corking the tube and setting it aside until the flocculent precipitate formed (of what?) settles, pipetting off an aliquot part of the clear supernatant solution and determining the N as ammonia present by comparing with a standard solution. Determinations made in this manner are reported by some analysts as "N as Direct Ammonia."

¹ Bartow and Harrison, J. Am. Chem. Soc., 32, 1256.

DETERMINATION OF TOTAL ORGANIC NITROGEN.

The determination of the total organic nitrogen in a water sample is seldom called for in a sanitary examination for the reason that the results are less easily interpreted than are those obtained from the processes giving Nitrogen as Free Ammonia and Nitrogen as Albuminoid Ammonia. In general the amount of nitrogen found as albuminoid ammonia multiplied by two will give an approximation of the total organic nitrogen present. Accurate determinations are important when dealing with sewage and sewage waters. These problems are, however, not considered in the present pamphlet. It is therefore sufficient to state that the process consists in applying a modification of the Kjeldahl method. For details see Jour. Infect. Diseases, 1905, May Sup. pp. 35-38, Amer. J. Pub. Hyg., 20, 363 (1910).

DETERMINATION OF "OXYGEN CONSUMED" OR
"REQUIRED OXYGEN."¹

By measuring the quantity of potassium permanganate reduced by a known volume of water, the treatment being under controllable conditions, an approximation of the amount and character of the organic matter present is possible, but it should be borne in mind in the interpretation of results that it is the carbon of the organic matter that is doubtless the chief reducing agent acting upon the permanganate.

In surface waters as has been shown by many investigators the amount of permanganate reduced is more or less proportional to the color of the water, and in ground waters usually bears a close relationship to the number of bacteria present.²

¹ Caldwell, et. al., J. Anal. Chem., 3, (1889).

² Chamot, Am. J. Pub. Hyg., 18, 280 (1908).

Process. 200 cubic centimeters of the sample are placed in a 400 cc. Erlenmeyer flask; a tiny fragment of ignited pumice stone and 10 cc. of dilute sulphuric acid (1:4) are added and from a burette standard potassium permanganate (1 cc. = 0.0001 gram of available oxygen) introduced very slowly, drop by drop. As soon as a permanent pink color results the burette reading is recorded, and the flask allowed to stand a few minutes so as to note whether decolorization will take place. The standard permanganate is again added until the total volume introduced is equal to 10 cc., the flask is then placed over a Bunsen or ring burner (best if heated over the free flame) and brought as rapidly as possible to a boil. The boiling is continued for exactly five minutes, the flask removed, allowed to stand for one minute, the excess of permanganate carefully destroyed by standard oxalic acid solution (1 cc. = 1 cc. permanganate) and the permanganate again added until a trace of pink appears and persists for one minute. From the total volume of permanganate the volume of oxalic acid used is deducted.

In order that the results obtained may always be comparable, an excess of at least 2 cc. must at all times remain.

Should the sample of water under examination destroy the 10 cc. of permanganate during the boiling, more permanganate is added until a permanent color results. A new sample of the water is now taken and sufficient standard permanganate added to insure an excess of at least 2 cc. after five minutes' boiling; an additional 5 or 10 cc. of the dilute sulphuric acid also being added.

Report all results in parts per million oxygen consumed.

Carried out in this manner the results obtained seem to be much more uniform than in most of the other modifications of this method; parallel determinations check and

results obtained on different samples of water are comparable.

The points of importance in this method of procedure are: (1) the slow addition of the standard permanganate solution in the cold to the acidulated sample; (2) the addition of an excess of permanganate before boiling and the insurance that an excess will remain after the period of boiling; (3) the presence of a decided excess of sulphuric acid at all times; and (4) that a period of boiling for five minutes is sufficiently long to yield all the information that an analyst needs.

The slow addition of the permanganate in the cold will cause the oxidation of nitrites, ferrous and manganous compounds, hydrogen sulphide, sulphites, thiosulphates, very easily oxidizable organic matter (usually putrefying animal matter) and unusual reducing substances. Many times this preliminary addition of the standard solution yields data of great value in the interpretation of results. Should the analyst wish an expression of the oxygen consumed due to stable organic matter, the amount of permanganate reduced at once in the cold is deducted from the total.

There appears to be no question that in the usual methods the organic matter present cannot be completely oxidized, even on very long boiling, and further that the amount oxidized bears no distinct relation to the total. As a rule, the analyst cares very little to know the exact quantity of very stable or resistant organic matter. The easily oxidizable unstable material interests him far more, and he can obtain all the information he wishes by boiling for not over five minutes. Long boiling unduly concentrates the sample, causes a decomposition of the permanganate irrespective of the organic matter,¹ and renders the obtaining

¹ See Weems and Brown. Influence of chlorine as chlorides in determination of oxygen consumed. Proc. Iowa Acad. Sci., 8, (1901).

of comparable results difficult and oftentimes impossible. The same reasons hold good for the use at all times of as nearly as possible a constant excess of standard permanganate, for it is evident that concentration plays a very important part in the quantity of the reagent reduced.

DETERMINATION OF NITRITES.¹

Process. Rinse very thoroughly with tap or nitrite free water (why not use distilled water?), two 100 cc. Nessler tubes. Pour into one of them 100 cc. of the sample to be tested; add 4 cc. of nitrite reagent, cork the tube (Why?) and allow to stand for half an hour. If a pink color develops compare it with the fuchsine standards of the laboratory and then make a comparison tube, using the proper amount of standard nitrite and nitrite free water, to which 4 cc. of nitrite reagent is then added. After standing half an hour pour the treated sample and the standard into colorimeter cylinders and read the intensities in the instrument as usual, from these readings calculate the amount of nitrogen as nitrite present in the sample.

Should the color developed in the sample be greater than 0.01 part per million, take 10 cc. of the sample, add 90 cc. of nitrite free water and proceed as directed above.

Water showing any color in a 100 cc. Nessler tube must be first decolorized with nitrite free "aluminum cream" before testing for nitrites. (Why?)

DETERMINATION OF NITRATES.

Before this determination is made it is necessary to ascertain the alkalinity, the chlorine and nitrite content, the color of the sample and whether much iron is present.

If the color is high decolorize with "aluminum cream."

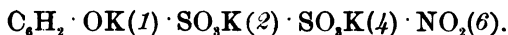
If the nitrites are in excess of one part per million of

¹ See Weston, J. Am. Chem. Soc., 27, 281 (1905).

nitrogen as nitrite they must be converted into nitrates as described below.

*Process.*¹ Measure into a porcelain evaporator 100 cc. of the sample of water or if the nitrate content is known to be very high, a smaller volume.

Add sufficient N/50 sulphuric acid calculated from the alkalinity to not quite neutralize all the alkalinity, being very careful not to make the sample neutral or acid (Why?). Then add such a volume of the silver sulphate reagent as will precipitate all but 0.1 mg. of the chlorine present. Heat to boiling (Why?), add a little aluminum cream (Why?), filter, wash the precipitate with hot water and evaporate the united filtrate and washings to dryness. Add to the dry residue 2 cc. of phenol disulphonic acid reagent, rubbing the entire mass with a glass rod to bring the reagent into intimate contact with the residue. Place the evaporator containing the residue thus treated on the water bath for a few minutes; cool, add about 50 cc. of distilled water and then very carefully with constant stirring a strong solution of KOH until the maximum yellow color is developed, then add about three drops in excess. The yellow color is due to the formation of tripotassium nitrophenol disulphonate, $C_6H_2OK(SO_3K)_2NO_2$, the disulphonic acid having one nitro group introduced into the molecule in position 6. The structural formula of the potassium salt is therefore



Filter into a colorimeter cylinder, dilute to the proper point and compare with a standard containing a known amount of nitrate.

To prepare the standard measure out from a burette into an evaporator the proper volume of standard potassium nitrate, add a little water, evaporate to dryness on the water

¹ Method of Chamot, Pratt and Redfield, J. Am. Chem. Soc., Mar. 1911.

bath, cover the dry residue with 2 cc. of sulphonic acid reagent, dilute, make alkaline, transfer to a colorimeter cylinder and dilute to the proper height. Compare the colors as usual.

In order to aid the student in ascertaining the proper amount of standard to evaporate, a series of tubes containing known amounts of tripotassium nitro phenol disulphonate will be found on the colorimeter table.

Should the nitrites in the water sample be over one part per million, an unheard of value, it is necessary to treat the water with sufficient dilute potassium permanganate to give a distinct pink color, heat on the water bath and when the pink color has disappeared add a little more permanganate, evaporate to dryness and proceed as usual. Subtract from the final result the amount of nitrogen due to the conversion of the nitrite present to nitrate (write equation for oxidation).

DETERMINATION OF PHOSPHORIC ACID AS PHOSPHATES.

Method of Woodman and Cayvan,¹ modified by Veitch.²

Process. Measure into a porcelain evaporator 50 cc. of the sample of water, add 3 cc. of dilute nitric acid (Sp. Gr. 1.07) and evaporate to dryness on the water bath, again add 3 cc. of nitric acid and again evaporate. Heat the residue for two hours in an air bath at the temperature of boiling water (Why?). Under no circumstances should the temperature rise above 100°C. (Why?).

Extract the dry residue with cold, freshly drawn distilled water in repeatedly added small amounts. Dilute to 40 cc. in a colorimeter cylinder, filtering if necessary. Distilled water which has been standing for some time in a glass bottle or flask should not be used (Why?).

¹ J. Am. Chem. Soc., 23, 96 (1901); 24, 735 (1902).

² J. Am. Chem. Soc., 25, 169 (1903); Bull., 31, U. S. Dept. Agr., Bur. Soils.

Add to the 40 cc. in the colorimeter cylinder 5 cc. of dilute nitric acid and 4 cc. of ammonium molybdate solution, fill to 50 cc., mix and compare with a known volume of standard sodium phosphate solution which has been diluted to 40 cc., and treated with 5 cc. of dilute nitric acid and 4 cc. of ammonium molybdate solution and diluted to 50 cc.

The treated cylinders should be allowed to stand for twenty minutes before the colors are compared.

Report all results in parts per million PO_4 .

Should it be necessary to determine the PO_4 with great accuracy, it is necessary to correct for soluble silica (Why?). For the correction process see foot note references 1 and 2.

In interpreting results it should be remembered that in some localities ground waters may, because of the geological formation which obtains, contain abnormally high amounts of PO_4 . This will also be the case when dealing with the run-off of cultivated fields recently treated with superphosphate fertilizers. As a rule, however, high phosphates in ground or surface waters indicate pollution of animal origin, as for example, sewage, barn-yard drainage, etc.

DETERMINATION OF SULPHATES BY COLORIMETRY.

Method of Bureau of Soils, U. S. Department of Agriculture, Bull. 31, page 49. (Modified Winkler Method).

"The method consists in treating the acidified solution to be examined with barium chromate, insoluble barium sulphate being formed, and upon neutralization, an amount of CrO_4 equivalent to the precipitated SO_4 remains in solution."

(Write all equations.)

Process. Measure into an Erlenmeyer flask 150 cc. of the sample (or less, if necessary), add about 10 drops of concentrated hydrochloric acid and sprinkle in from 0.1 to 0.2 gm. of pure barium chromate. Rapidly bring the contents

of the flask just to a brisk boil. Cool at once, and then, to the completely cooled liquid, sodium hydroxide solution (1:10) is carefully added, until a tiny drop of the liquid placed upon red litmus paper produces a blue color; a greater excess of alkali must be avoided. Filter through a filter of close texture, pouring back the first runnings, if cloudy. Transfer 200 cc. of the clear filtrate to a colorimeter cylinder.

To prepare the standard for comparison, pour 80 cc. of distilled water into a colorimeter cylinder, add a few drops of sodium hydroxide solution and introduce from a burette sufficient standard dichromate solution to match the color of the sample treated as above described. Fill up to the 100 cc. mark with distilled water, mix and compare in the Wolff colorimeter.

Owing to the solubility of barium chromate a correction must be applied. This correction for solubility has been found to be equivalent to 8.2 parts of SO_4 per million of solution. Report all results in parts per million SO_4 .

It is also possible to determine the amount of SO_4 present in a water with a fair degree of accuracy by comparing in a colorimeter the amount of turbidity produced by barium chloride after acidulation with hydrochloric acid, with that produced in a standard solution of potassium sulphate, treated in like manner. (Method of Belz. Bull. 31, Bureau Soils, page 54).

DETERMINATION OF IRON.

*Process.*¹ Total Iron.

Evaporate 100 cc. of the sample to dryness in a porcelain evaporator on a water bath. Add a few cubic centimeters of aqua regia and tilt the dish so as to cause the acid to flow over the entire residue. Evaporate again to dryness IN THE HOOD. After cooling add 5 cc. of dilute HCl, tilt-

¹ Sutton, Volumetric Analysis.

ing so as to moisten the whole residue. Dilute with distilled water, warm and filter into a 100 cc. measuring flask or Nessler tube, wash the filter paper with repeated small volumes of distilled water until the liquid almost fills the container, add a drop or two of dilute permanganate solution (Why?), allow to stand until the pink color disappears (Why?), add 2 cc. of ammonium sulphocyanate reagent, fill to the mark with distilled water and transfer to a colorimeter cylinder. *or ferrous*

Compare the color with that obtained by treating a known volume of standard iron solution with a like volume of sulphocyanate reagent.

Should the sample be high in iron or high in iron containing suspended matter, less than 100 cc. should be taken for the determination.

If much organic matter is present, gently ignite the residue after the first evaporation. (Why not ignite after treating with aqua regia?)

Report results in parts per million Fe.

It is probable that the iron is present in most waters in colloidal condition, less often as ferrous carbonate (bicarbonate?) or as a ferric alum.

DETERMINATION OF MANGANESE.

Method of Weston.¹

Process. "Evaporate with about 25 cc. of nitric acid (Sp. Gr. 1.135) enough of the sample to give from 0.01 to 1. mg. of manganese. Gently ignite the residue or bake it for half an hour at 130°C. Add 50 cc. of nitric acid (1.135) and when the solution is cool add about 0.5 gm. of sodium bismuthate" (Why? Write equations.). "Heat until the pink color disappears." (Why?)

If a black or brown precipitate appears clear the solu-

¹ J. Am. Chem. Soc., 29, 1074 (1907).

tion by the cautious addition of sodium thiosulphate (Why?)

“To the cool solution free from oxides of nitrogen, add sodium bismuthate in excess, stir a few minutes, and filter through thoroughly washed asbestos in a Gooch filter. Wash with dilute nitric acid, transfer filtrate to a large Nessler tube and make up to 100 cc. with dilute nitric acid. In another tube put about 100 cc. of dilute sulphuric acid and add standard potassium permanganate (1 cc. = 0.0001 gm. Mn) until the color of the sample is matched.”

Report all results in parts per million Mn.

The method is sufficiently accurate for the requirements of ordinary water analyses.

Few waters contain an appreciable amount of manganese but occasionally manganese impregnated ground waters are met with.

DETERMINATION OF CHLORINE AS CHLORIDES.¹

Process. Measure 100 cc. of the sample into a No. V. evaporator; add 1 cc. of potassium chromate solution (freed from chlorides). Titrate with standard silver nitrate, using a small burette graduated in 1/20 cc. and wearing at the time goggles with yellow glass or performing the titration under yellow light. Observe the same precautions and follow the same method as that employed in the standardization of the silver solution (see page 13).

The most accurate results are obtained when the volume of silver nitrate employed is approximately the same as that used in standardizing and the final volume of the water sample titrated corresponds to that obtained in adjusting the strength of the silver solution.

If, therefore, it is found advisable to employ less than 100 cc. of the water sample first add sufficient distilled water to make 100 cc.

¹ Hazen, Am. Chem. J., (1889) p. 409.

Should the volume of silver solution used fall much below that employed in standardizing add to the portion of the sample to be titrated such a measured volume of standard sodium chloride as will raise the volume of silver to be used to the proper value, subtracting this equivalent from the final reading.

It is believed that small quantities of copper salts present in a water may introduce a serious error in the chlorine determination, a fact well to bear in mind in view of the extensive use of copper sulphate as an algicide.

Such waters give, when titrated as above, an end point which vanishes in a few seconds. The addition of another drop of silver nitrate solution gives a red color rapidly fading away.

The most reliable results are obtained when the volume of standard silver nitrate used is more than 1 cc. and less than 10 cc.

In the case of waters very high in chlorides determine the chlorine gravimetrically just as in standardizing hydrochloric acid.

DETERMINATION OF ALKALINITY.

Solutions required, N/25 or N/50 sulphuric acid.

Indicator, erythrosin, ethyl or methyl orange.

Para nitrophenol, phenacetolin, lacmoid and sodium alizarin sulphonate have also been suggested. Of these phenacetolin has a very uncertain end point, while lacmoid and alizarin compounds require boiling of the water being titrated (Why?).

Process. Applicable to all waters.

Into a 150 cc. glass stoppered bottle introduce 100 cc. of the sample, add 2 cc. of erythrosin indicator, 5 cc. neutral chloroform (Is the chloroform of the laboratory usually acid or alkaline? Why?). Shake well. If the water is alkaline it will be colored pink. Run in from a burette a

very small volume of N/50 sulphuric acid, insert the stopper of the bottle and shake, continue this process of adding acid and shaking, proceeding with great care toward the end of the titration and allowing the titrated water to stand a few seconds after each shaking. The end point is reached when the pink color of the water above the chloroform layer has just been discharged. Look through this layer sideways through the bottle and disregard the color in the chloroform layer.

The "end point" with erythrosin is usually very sharply defined. It is the only indicator which can be safely used when dealing with waters turbid with clay or other aluminum compounds in suspension or when aluminum or iron compounds are believed to be in solution or suspension, as, for example, in the effluents of filters of the "American" or "Mechanical" type.

Alternate Process. Applicable to waters not containing clay in suspension or iron or aluminum compounds in moderate amount.

A. With Methyl or Ethyl Orange or Para Nitrophenol.

Into a 200 cc. Erlenmeyer flask pour 100 cc. of the sample. Add just enough indicator to give a distinct color. It is always best to have indicators of definite strength so that the same volume will be used in the sample as was employed in adjusting the standard solution. (Why?). It is therefore customary in the Cornell Laboratory to make the indicator of such strength that 1 cc. may be measured out and employed.

Grasp the flask by the neck in the right hand, and, giving it a whirling rotary motion, run in N/50 sulphuric acid by operating the stopcock of the burette with the left hand. Run in the standard acid rapidly at first, then drop by drop, until the color just changes from alkaline to neutral (or the same tint employed in standardizing). The end

point should be reached without having stopped the titration or set down the flask.

B. With Lacmoid or Sodium Alizarin Sulphonate.

Pour 100 cc. of the water into a 200 cc. Erlenmeyer flask. Add 0.5 cc. of the indicator. Run in the standard N/50 H_2SO_4 rapidly from a burette, being careful to stop considerably before the end point is reached. (Why?). Heat (Why?) rapidly to brisk boiling over a free flame, giving the flask a rotary motion. A band of folded filter paper around the neck of the flask will permit the holding of it without burning the fingers. Add more acid and again boil. This process is kept up until the first change of color appears. Usually, watching the last drops of acid as they fall through the liquid will give a better idea of the end point than looking for a change in the color of the whole liquid.

DETERMINATION OF MINERAL ACID HARDNESS.

The results obtained by the process described below were formerly reported as "Incrustants" or as "Normal Hardness" and approximate the "Permanent Hardness" as reported by chemists some years ago.

Methods of American Public Health Association.

Process. "Measure 200 cc. of the water into a Jena glass Erlenmeyer flask (or flask of other resistance glass); boil ten minutes to expel free carbonic acid (Why?), and add 25 cc. of N/10 soda reagent (q.v.). Boil to a volume of 100 cc. (Why?) and cool (Why?), rinse into a 200 cc. graduated flask and make up to 200 cc. with boiled (Why?) distilled water. Filter, rejecting the first 50 cc. (Why?) and titrate 100 cc. of the filtrate for excess of soda reagent, using N/10 sulphuric acid with erythrosin and chloroform as an indicator. If a blank determination is made the difference between the number of cubic centimeters of sul-

phuric acid used in the blank and the test gives the mineral acid hardness directly."

"When the number of cubic centimeters used in the tests exceeds the number used in the blank, carbonates of sodium or potassium are indicated."

It then becomes necessary to determine the calcium and magnesium present as carbonates (see Examination of Water for Steam Generators, p. 91), compute the alkalinity due to these compounds and subtract from the total alkalinity. All hardness and alkalinity are always reported in parts per million CaCO_3 .

TOTAL HARDNESS BY SODA REAGENT.

Having first determined the alkalinity with N/50 sulphuric acid and erythrosin, add to 200 cc. of the sample the exact volume of N/50 sulphuric acid required to neutralize the alkalinity. From this point proceed exactly as in the determination of incrustants; Boil down, add the soda reagent, boil, cool, dilute to original volume with boiled distilled water, filter, rejecting the first running, and titrate 100 cc. of the filtrate with N/10 acid.

The Total Hardness minus the alkalinity = the "Mineral Acid Hardness" or "Permanent Hardness."

DETERMINATION OF ALKALINITY DUE TO NORMAL CARBONATES OR DUE TO CAUSTIC ALKALIES OR LIME ("HYDRATES").

"Normal carbonates are present in a water when the phenolphthalein alkalinity in the cold is equal to or less than one-half of the alkalinity by erythrosin."

When titrating with N/50 H_2SO_4 with phenolphthalein in the cold how much of the normal carbonate alkalinity is indicated? Why?

If only normal carbonates are present in the water being titrated with H_2SO_4 , what will be the relation between

the phenolphthalein alkalinity and the erythrosin alkalinity?

When phenolphthalein gives no color with the water in the cold what is indicated?

“When waters contain calcium or other alkaline hydrate as well as normal alkaline carbonates, the phenolphthalein alkalinity is more than one-half that with erythrosin.” (Explain fully.)

To determine the hydrate alkalinity first “Determine the alkalinity with phenolphthalein in the cold with N/50 H_2SO_4 and the total alkalinity with erythrosin. The alkalinity by the former indicator subtracted from that by the latter and multiplied by two gives the normal carbonate alkalinity; this subtracted from the total alkalinity gives the hydrate (or caustic) alkalinity.” (Explain fully.)

Supposing that the alkalinity of a water is due to caustic alone, what will be the relation of the phenolphthalein alkalinity to that by erythrosin?

Determination of “Hydrate Alkalinity” becomes of paramount importance in water purification or water softening plants where caustic alkalies or lime are employed in the process.

DETERMINATION OF ACIDITY.

The acidity of a water may be due to (a) free carbon dioxide, (b) free mineral acid, generally sulphuric, (c) acid salts, (d) aluminum sulphate (“filter alum”) used in excess in water purification, (e) iron sulphate, ferrous or ferric, excessive use in water purification or in mine waters, (f) organic acids or acid reacting organic compounds.

To 100 cc. (or less) of the sample, add 1 cc. of phenolphthalein indicator and titrate with standard sodium carbonate. There is thus obtained the acidity due to free CO_2 , free mineral acid, acid salts, salts of aluminum and iron and organic compounds = total acidity.

To 100 cc. of water (or less) add methyl or ethyl orange indicator and titrate with sodium carbonate = "free mineral acid acidity," reported in terms of SO_4 .

To 100 cc. of water (or less) add erythrosin indicator (and chloroform) and titrate with sodium carbonate = acidity due to "free mineral acids" and to sulphates of iron and aluminum.

In another portion of the sample determine the amount of iron present both in ferrous and ferric condition. Determine the aluminum present and the total SO_4 . From all the above acidity data and these results it is possible to calculate the acidity due to CO_2 , free mineral acid, and sulphates of iron and aluminum.

In effluents from purification plants using filter alums, if the reaction of the water is not alkaline to erythrosin, titrate with standard sodium carbonate, (N/50).

Determination of acidity due to organic compounds is rarely possible. In practice it is customary to determine only free carbon dioxide, acidity due to sulphates of iron and aluminum, and call all the remainder "mineral acid acidity," reported in parts per million SO_4 .

When the water is to be used for boiler or other industrial purposes it is becoming more and more the practice to report acidity in terms of hydrogen ions. Factors for such conversions will be found in the tables given in the appendix, page 110.

DETERMINATION OF HARDNESS BY MEANS OF SOAP SOLUTIONS.

Methods based upon the decomposition of soap by carbonates and other salts of calcium and magnesium in solution in water were formerly in general use, but have been shown in recent years to be so unreliable that the process will not be here described. The results are without value when waters contain carbonates of sodium or potassium or their hydroxides, or if calcium hydroxide or much mag-

nesium are present. It is therefore evident that the method is restricted to comparatively soft waters in which there are in solution only small amounts of the carbonates of calcium and magnesium alone.

DETERMINATION OF CARBON DIOXIDE.

Carbon dioxide is present in waters as free carbon dioxide in solution and in combination with bases as primary carbonates, now termed "carbon dioxide as bicarbonates," formerly reported as "half-bound" carbon dioxide; and in combination with bases as normal carbonates, formerly reported as "Fixed carbon dioxide."

DETERMINATION OF FREE CARBON DIOXIDE.

Method of Seyler, as modified by Elms.

*Process.*¹ Pour 100 cc. of the sample into a 200 cc. Erlenmeyer, add 1 cc. of neutral phenolphthalein indicator and run in, from a burette or graduated 10 cc. pipette, N/22 sodium carbonate solution as rapidly as possible, giving the flask a rotary motion, but avoiding any splashing of the sample (Why?). Stop at the appearance of a very faint pink which remains permanent for a few seconds. The pink color soon disappears on standing (Why?).

Somewhat more accurate results may be obtained after a little practice by placing the sample in a 100 cc. Nessler tube, adding the indicator and running in the standard sodium carbonate, the solution being stirred by means of a glass plunger, the first appearance of a pink color being determined by looking down through the tall column of liquid. The flask method is more rapid, gives less trouble and yields results sufficiently accurate to meet all ordinary requirements.

¹ Journ. Infec. Dis., 1905, May Sup.

The free carbon dioxide content of a water can be determined accurately only when determinations are made in the field. When this cannot be done it is essential that the sample bottle shall be filled so full as to leave no air space and that the sample thus obtained shall be kept cold (on ice) until the determination is made.

DETERMINATION OF CARBON DIOXIDE AS NORMAL CARBONATES
AND BICARBONATES.

*Method of Cameron.*¹

Process. To 100 cc. of the water contained in a 250 cc. Erlenmeyer flask add 1 cc. of phenolphthalein indicator. If normal carbonates are present, the solution will have a red or pink color. Run in, drop by drop, from a burette, a standard solution of H_2SO_4 , allowing a few seconds to elapse after each addition of standard solution, continuing until the red color just disappears. Record the burette reading and add to the flask 1 cc. of methyl or ethyl orange indicator. Continue the titration (without refilling the burette) with the standard sulphate solution until the indicator shows a change in color. This end point may be determined if a second flask treated just as the first is placed side by side with that being titrated. The reading obtained with phenolphthalein is that due to normal carbonates.



Write the equation for the next step in the titration (with methyl orange) and explain how the CO_2 as bicarbonates present may be calculated.

The amount of bicarbonates and normal carbonates present may also be ascertained by the following rules.²

¹ Bull. 91, p. 19, Div. of Chem., U. S. Dept. Agric. and Bull. 31, Div. of Soils.

² Methods of Water Analysis, Amer. Pub. Health. Assoc., J. Infec. Dis., May Sup. 1905, and Amer. J. Pub. Hyg., June 1910.

“When water is acid to phenolphthalein, the CO_2 as bicarbonates is equal to 88 per cent. of the alkalinity when the latter is expressed in terms of CaCO_3 ”. (Explain why.)

“Carbonic acid as bicarbonates is present only when the alkalinity by phenolphthalein is less than one-half of that by erythrosin (or lacmoid)”. (Derive the proper formula for computing, if n = number of cc. of $\text{N}/50$ H_2SO_4 required when phenolphthalein is used and m = number of cc. of $\text{N}/50$ acid used in determining the alkalinity of 100 cc. of water with erythrosin.)

Carbonic acid as normal carbonates is computed “in parts per million of CO_2 as 44 per cent. of the alkalinity by erythrosin in terms of CaCO_3 minus one-half the CO_2 previously determined as bicarbonates.” (Explain fully.)

DETERMINATION OF TOTAL SOLID RESIDUE.

The Total Solid Residue of a water is obtained by the evaporation of a known volume of the sample to dryness on the water bath and subsequently heating to constant weight. This value gives the sum of the solids in solution and solids in suspension.

*Process*¹. Shake the sample very thoroughly in order to obtain an even distribution of the material in suspension and pour out *immediately* 100 cc. into a measuring cylinder, transfer to a weighed platinum dish and evaporate to dryness on a water bath, rinsing the measuring cylinder with a little distilled water to absolutely insure the transference of all suspended matter which might have settled out. As soon as the contents of the dish are dry, place in an air bath at 100° to 105°C . or in a Victor Meyer bath heated by toluene. After about thirty minutes, cool in a desiccator, weigh and return to the air bath; as soon as constant weight is obtained record the results as parts per million of total solid residue.

¹ Method American Public Health Association.

When much magnesium is present it is next to impossible to obtain correct weighings (Why?), under such conditions it is best to add to the water being evaporated an amount of standard sodium carbonate solution equivalent to 25 cc. of N/50 sodium or potassium carbonate solution, correcting the final weighings for the amount of material added.

After weighing the total solids place the dish on a pipestem triangle and holding a Bunsen burner in the hand play the flame over the bottom and sides of the dish in such a manner as to heat dish and contents to a *very low* red heat, noting well the change in appearance and color of the residue and any odors given off. Cool, weigh, heat again and again weigh. Continue until the weight is approximately constant. The residue should now be white or nearly so. Moisten with a few drops of distilled water, dry on the water bath and heat in an air bath to constant weight as in the case of the total solid residue. This value gives the "Fixed Solids," which on subtraction from the total solid residue gives *loss-on-ignition*.

In the case of water analyses with reference to steam generation it is the custom of many analysts to attempt an approximation of the organic matter present by allowing the moistened ignited residue to remain for some hours in an atmosphere of carbon dioxide (Why?) drying and weighing. The difference between the weight of total solids and the CO₂ treated residue is reported as "Organic matter." (Why is this supposed to give a more accurate indication of organic matter than loss-on-ignition?)

Solids in Suspension. Filter 150 to 200 cc. of the thoroughly shaken sample through a small Berkefeld (or Garros) filter, or use the filtrate employed in the determination of color. These filtering tubes are of sufficiently close texture to remove the finest silt carried by a water yet at the same time remove considerably less of the dissolved

solids than tubes of porous porcelain of the Pasteur-Chamberland type. Always reject the first portion of the filtrate (Why?). Measure 100 cc. of the clear filtrate into a weighed platinum dish and proceed exactly as directed for the determination of total solid residue. The increase in weight over the weight of the empty dish will give the solids in solution. This value subtracted from the total solids gives "solids in suspension."

Coefficient of Fineness. Divide the weight of the solids in suspension expressed in parts per million by the turbidity of the sample. The quotient thus obtained is known as the coefficient of fineness of the suspended matter. If greater than unity the suspended matter is coarser than the fuller's earth turbidity standard, and if less than unity it is finer than the standard.

DETERMINATION OF DISSOLVED OXYGEN.

Method of Levy, Mont-Souris Observatory of Paris.¹

I. Carefully determine the exact volume of boiled distilled water at 20°C. held by the Romyn pipette between the stopcocks.

II. Just before a determination is to be made prepare 25 cc. of a solution of ferrous ammonium sulphate of such strength that one cc. will require approximately 0.0003 gm. of oxygen for oxidation. Titrate an exactly measured portion of this solution with standard potassium permanganate solution (that prepared for determination of oxygen consumed). What volume of the ferrous ammonium sulphate should be employed? Record in the note book the weight of the iron salt to be used since a fresh solution must be prepared and standardized just prior to each analysis.

When, however, many determinations are to be made a stock solution of the iron salt may be prepared provided it is slightly acidified with sulphuric acid (Why?). A portion

¹ Ann. de l'Observatoire de Mont-Souris, 1883.

must be titrated with permanganate however, just before being used. (Why?)

Process. Fill the pipette by immersing it, with both stopcocks open, in the water to be examined. If, however, the sample is contained in a bottle proceed as follows. Attach to the lower opening a piece of rubber tube, to the opposite end of which a small funnel is attached, fill the tube with the water and raise the funnel until the water just enters the cylindrical portion of the pipette. Close the lower stopcock, remove the funnel, compress the end of the rubber tube with the fingers and introduce it into the sample container; lower the pipette, still holding vertically until the lower end is slightly below the level of the water in the sample bottle, open the stopcocks and allow the water to slowly siphon into the pipette, always avoiding a rapid flow (Why?). When full, close both stopcocks, noting that the bores of the stopcocks are filled with water; empty both tubes, fill the lower one full with freshly boiled distilled water (Why?), hold a finger over the end, being careful to entrap no air (Why?) and invert into a small evaporator containing a few cc. of freshly boiled distilled water, to which 0.5 cc. of dilute sulphuric acid is then added (Why?). Clamp the pipette in a vertical position. Introduce into the upper tube 1 cc. of the standardized ferrous ammonium sulphate solution, avoiding air bubbles. Open the upper stopcock, then very cautiously open the lower stopcock of the pipette, thus drawing in all the iron solution, but without introducing any air either into the pipette or into the bore of the stopcock. Close both stopcocks. Remove the pipette from the clamp and mix by inverting a few times until the liquid appears homogeneous (Why?). Wash out the upper tube (Why?). Replace the pipette in its clamp (after filling the lower tube with boiled water) so that the lower tube again dips into the acidulated water in the

evaporator. Pour 1 cc. of the caustic alkali solution, (page 24), into the pipette tube and introduce it into the pipette (Why?) in the same manner as the iron solution, observing like precautions, close both stopcocks, remove from the clamp and mix thoroughly. After standing for about half an hour (Why?) fill the upper tube with the sulphuric acid solution. (page 24) Open the upper stopcock only, and allow to stand until all the precipitated iron hydroxide is dissolved (Why?). Empty the pipette into an Erlenmeyer flask, rinse with freshly boiled distilled water, adding the rinsings and the contents of the evaporator (Why?) to the solution in the Erlenmeyer. Add 10 cc. 1:4 sulphuric acid. Titrate with standard permanganate, thus ascertaining the quantity of ferrous iron not oxidized by the oxygen in the water. The difference between the two permanganate titration values gives the quantity of ferrous salt oxidized by the oxygen dissolved. In calculating the results what correction of the pipette volume must be made in order that the exact volume of the sample treated may be determined? Write all the reactions involved in the method.

Report results in parts per million of oxygen and also in cubic centimeters of oxygen gas per liter at 0°C. and 760 mm. pressure. Not infrequently it is also desirable to ascertain whether the amount of oxygen present in the sample has reached the saturation point or what per cent. of saturation exists. According to Winkler¹ water saturated with air at 760 mm. pressure contains the following quantities of dissolved oxygen in parts per million.

¹ Ber., 22, 1772 (1889).

Temperature C°.	Oxygen	Temperature C°.	Oxygen
0	14.70	14	10.35
1	14.28	15	10.14
2	13.88	16	9.94
3	13.50	17	9.75
4	13.14	18	9.56
5	12.80	19	9.37
6	12.47	20	9.19
7	12.16	21	9.01
8	11.86	22	8.84
9	11.58	23	8.67
10	11.31	24	8.51
11	11.05	25	8.35
12	10.80		
13	10.57		

Occasionally waters are met with in which the dissolved oxygen found is in excess of the saturation shown in the table.²

Note. The Levy method is comparatively little used in America, requiring as it does a special form of apparatus and being somewhat less exact than that known as the Winkler method; however, the Levy method has the advantage both of requiring no reagents nor standard solutions not used in some other water analysis process and of being somewhat simpler.

The Winkler method consists in adding to the sample, contained in a glass stoppered bottle, manganous sulphate solution, followed by an alkaline solution of potassium iodide, and finally sulphuric acid. The final titration is made with a standard solution of sodium thiosulphate with starch as indicator.

Write all the reactions involved in this method.

² Lord, Sources of Water Supply of Ohio. Special Rept. State Board of Health, 1897-98, page 53.

For specific details as to the standard of procedure in the Winkler method, as adopted by the American Public Health Association, see May Supplement, 1905, Journal Infectious Diseases.

In connection with the determination of dissolved oxygen see page 89. Estimation of Putrescibility.

TEMPERATURE.

Temperature measurements should be made in the field by means of some type of electric resistance thermometer if possible.

"Bucket thermometers" rarely give readings of sufficient accuracy to be of value if temperatures at different depths are wanted, since the "bucket" is generally too small to maintain the temperature of the level wished during the time the instrument is being raised for reading. A better plan is to enclose a short thermometer within a bottle provided with a weight and device for pulling the stopper when the proper depth is reached. The bottle thus prepared is stoppered and lowered into the water to the depth desired, allowed to remain there for a short time for the glass to acquire a temperature equilibrium, the stopper pulled, thus filling the bottle with a sample of the water at that level, then after a few minutes the apparatus is rapidly raised to the surface and the thermometer read at once while still completely immersed.

In the case of well tubes so small as to forbid the use of the above methods, pump slowly for a sufficiently long time to cool, or warm, the pipes to the water temperature, then collect a pailful; completely immerse the thermometer and as soon as the mercury column becomes stationary read the instrument.

If an analyst makes it a point to obtain whenever possible the temperature of the sample of water to be examined he will find that in a very large number of instances this

data will yield information of the greatest value in properly interpreting the results of his analyses.

APPEARANCE.

The analyst should record in his note book the characteristics exhibited by the sample when viewed in a bottle of clear white glass. The appearance of a water is dependent upon the materials in solution and in suspension, gases in solution, etc. The following adjectives are usually employed as descriptive of the appearance: sparkling, limpid, bright, clear, cloudy, murky, milky, muddy, etc.

ODOR.

Observations of the odor of the water sample should be made in the field, if possible, as well as in the laboratory. Immediately upon removing the stopper from the bottle containing a sample for chemical analysis observe the odor. After taking out the portions for the determinations of "Free Carbon Dioxide" and "Oxygen Dissolved," pour a portion into a clean, odorless, 500 cc. wide-mouthed glass stoppered bottle until the bottle is about two-thirds filled. If the sample is very cold allow it to stand until it reaches room temperature. Shake violently, remove the stopper and observe the odor at once. Record as "Cold odor."

Place the bottle in a water bath and after a few seconds warming to allow for air expansion insert the stopper. After heating for ten to fifteen minutes, remove the bottle, wrap it in a towel, shake, remove the stopper and cautiously smell the odor given off. Record as "Hot odor."

In expressing the quality of the odor follow as closely as possible the descriptive terms and abbreviations—

v = vegetable

a = aromatic

e = earthy

f = fishy

g = grassy

m = mouldy

M = musty

d = disagreeable

p = peaty

s = sweetish

In special cases express the odor by employing the best descriptive term applicable.

The intensity of the odor is indicated by a numerical value in accordance with definitions first suggested by the New England Water Works Association and now in general use.

"Numerical

Value	Term	Definition
0	None	No odor perceptible.
1	Very faint	An odor that would not be ordinarily detected by the average consumer, but could be detected in the laboratory by an experienced observer.
2	Faint	An odor that the consumer might detect if his attention were called to it, but that would not otherwise attract attention.
3	Distinct	An odor that would be readily detected and that might cause the water to be regarded with disfavor.
4	Decided	An odor that would force itself upon the attention and that might make the water unpalatable.
5	Very strong	An odor of such intensity that the water would be absolutely unfit to drink (a term to be used only in extreme cases)."

In accordance with this system of expressing and recording results, a water reported as—odor 4 f—would be one possessing a "decided fishy odor," objectionable to most consumers.

Observations of odors must never be omitted since in most instances when an odor may be detected an indication

is usually thus obtained as to the character of the plankton of the water or of the nature of polluting or contaminating material.

In many instances, shaking from 500 cc. to 1 liter of the water with a small amount of ether, separating the ether and allowing it to evaporate at room temperature and observing the odor of the residue, will yield data of much value. The odor given off by the residue being more concentrated and often better identified. Unfortunately such a procedure is rarely possible for want of sufficient sample.

COLOR.

By the term color is to be understood the "true color" due to substances in solution only, and observation of the color may therefore be made only upon a sample free from suspended matter or after the removal of suspended matter by filtration. An observation made upon the unfiltered water gives the appearance of the sample (Apparent color).

The color of natural waters in the eastern United States is almost invariably some shade of yellow or brown, with either a tinge of red or green. The tint is almost exactly that given by Nessler reagent with ammonia salts, and it was customary for many years to record the intensity of color in terms of the equivalent of parts per million, nitrogen as ammonia. It was pointed out some years ago by Hazen that this same tint could be duplicated by a mixture of platinum and cobalt salts and he therefore proposed expressing all measurements of color in terms of parts per million metallic platinum.

Within the last few years this so called Platinum-Cobalt Standard has been generally adopted.

The standard solution is thus prepared:—"Dissolve 1.246 grams of potassium chloro-platinate (K_2PtCl_6 , the weight given contains 500 mg. Pt.) and 1. gram of crystallized cobalt chloride ($CoCl_2 \cdot 6 H_2O$) in 500 cc. of water to

which 100 cc. of concentrated HCl has been added. Dilute to 1 liter in a measuring flask." The standard color solution thus prepared has a color of 500.

From the stock standard solution dilutions are prepared having colors of 5, 10, 15, 20, 25, 30, 35, 40, 50, 60, and 70. This series is best kept in 200 cc. glass stoppered bottles.¹ For comparison with water samples, portions of the properly diluted standard are poured into *dry* 100 cc. Nessler tubes or into *dry* colorimeter cylinders.

Determination of Color.

If the sample does not appear clear when viewed lengthwise in a 100 cc. Nessler tube, filter 200 cc. through a clean Berkefeld or Garros filter tube, rejecting the first 25 to 50 cc. of the filtrate. Tubes or crucibles made of porous porcelain or alumina (Pasteur-Chamberland, Alundun, Maasen, etc., types) may not be employed because of their marked adsorptive properties, thus leading to results which are too low. The Berkefeld tubes have also more or less adsorptive properties, so that the colors of filtered samples are usually from one to five parts per million too low. A very small amount of suspended matter in the sample causes a relatively great error by apparently increasing the intensity of the color. Transfer a portion of the clear filtrate to a tall 100 cc. Nessler tube or colorimeter cylinder. Compare with the color standards described above. "The color of a sample shall be observed by filling a standard Nessler tube, to the height equal to that in the standard color tubes, with the water to be examined. The observation shall be made by looking vertically downward through the tubes upon a white surface."¹

¹ Such a series will be found upon the laboratory reagent shelves, hence neither standard nor dilutions need to be prepared by the students.

¹ Standard Methods of Water Analysis. American Public Health Association.

Should the color of the sample be found to be greater than 70, dilute with distilled water.

After determining the color of a filtered sample preserve the filtrate for the determination of solids in suspension, see page 54.

The results of color observations are to be expressed in whole numbers as follows.

“Color between 1 and 50 recorded to nearest unit.

Color between 51 and 100 recorded to nearest 5.

Color between 101 and 250 recorded to nearest 10.

Color between 251 and 500 recorded to nearest 20.”

Determination of Color with U. S. Geological Survey Color Tubes and Glasses.

“*Color Standards.* Disks of colored glass have been prepared as standards for measuring color of water in the field. These disks have been rated to correspond with the platinum-cobalt standard. The color is measured by balancing the color of the water in a metallic tube with glass ends against the colors of glass disks of known value. The number on each disk represents the corresponding color of a water. Each disk carries two numbers. One is over 100, and is a serial number for the purpose of identification. The other number is less than 100, and shows the color value of the disk; that is to say, the color of each disk is equal to the color of a solution of the designated number of parts per million of platinum with the required amount of cobalt to match the hue when seen in a depth of 200 mm. When a water comes between two disks its value can be estimated between them by judgment. Two or more disks can be used, one behind the other, in which case their combined value is the sum of the two individual values. This is not always true, but the error introduced by adopting as the color of the combined disks the sum of their values, is usually negligible. By combining the disks of a series in different ways

a considerable number of values can be produced, allowing the closer matching of many waters."

"Filling the tubes. The tube, having an aluminum stopper, is to be filled with water, the color of which is to be determined. If the tube is not dry rinse it once or twice by filling and emptying it. The second tube, having the clips to hold the glass disks, is made much like the one holding the water, to facilitate comparison. Theoretically, this tube should be filled with distilled water. Practically, it makes very little difference whether it is filled with distilled water or empty. Use distilled water when it is convenient to do so, and when distilled water of unquestionable quality is at hand; otherwise wipe the inside of the tube dry to prevent fogging of the glass ends, and proceed with the tube empty."

"Holding the tubes. Hold the tubes at such a distance from the eye that the sides of the tubes just cannot be seen. This occurs when the near end of the tube is eight or nine inches from the eye. Hold the tubes at such an angle that both can be seen at once with one eye. Good results cannot be obtained in any other way. Let the tubes change places once or twice, as sometimes the light on the right and left is not quite equal."

"Background. There should be a clear white background with a strong illumination. The best results cannot be obtained with either too little or too much light. On a gray day look at the sky near the horizon and away from the sun. On a bright day look at a piece of white paper or tile upon which a strong light falls. The white surface may be vertical and the tubes held horizontally, or the tubes may be held at an angle directed downward toward a horizontal surface, as may be most convenient. Good results cannot be obtained by artificial light."

"Turbid Water. The colors of very turbid waters

cannot be measured in this way. Slight turbidities do not interfere seriously with the results. Waters too turbid for direct observations should be filtered through thick filter paper before being tested; and in case the suspended matter causing the turbidity is fine in grain and large in amount, even this method may fail. The turbidity of water to be taken as far as possible in connection with color observations, except in cases where it is obvious from inspection that there is practically no turbidity."

"Highly colored waters. Some waters will be found having a higher color than can be matched by the standards. In general, waters with colors above 100 should not be matched in 200-mm. tubes, and the results with waters having colors below 80 will be considerably more accurate than with more highly colored ones. Two procedures are possible with waters having higher colors; namely, to dilute with distilled water before measuring the color, or to use a shorter tube. The latter procedure is the more convenient, but both are equally accurate. To measure the color with short tubes, put the highly colored water in a tube of one-half the usual length and match as usual. It is not necessary to have a short standard holder. The 200 mm. tube can be used. After the water is matched the result is multiplied by 2. In case the color is too high to be read in a 100-millimeter tube it can be put in a 50-millimeter tube, and the result multiplied by 4. When dilution is used the highly colored water is mixed with one or more volumes of distilled water, the color is matched, and the result multiplied by a corresponding factor. The tube itself can be used for measuring the colored water and the distilled water, and the mixing can be done in a tumbler or any other convenient clean vessel."

"Cleaning the Tubes. Always keep the tubes clean. Take particular care of the glass ends. All the ends are

removable for the purpose of cleaning, and should not be screwed on too tightly. They should be water tight when screwed up loosely, for if screwed on hard, they may stick so as to come off with difficulty."

The above directions are quoted from Circular 8, U. S. Geol. Survey, Div. Hydrography.

DETERMINATION OF TURBIDITY.

Much time and thought has been devoted in recent years by sanitary engineers and analysts to studies on the turbidities of waters and to the method for ascertaining and recording the character and amount of material carried in suspension by water.

In problems involving the designing and operating of water purification plants, the amount, character and seasonal occurrence of the turbidity of the water to be treated are questions of paramount importance. But the student should not gain the impression that it is only when dealing with water purification that a record of turbidity is essential, for many a problem in sanitary water analysis arises in which determinations of turbidity are of inestimable aid in properly interpreting other analytical results.

Turbidity standard.

In the new standard proposed by the Laboratory Section of the American Public Health Association, precipitated fuller's earth is substituted for the silica prepared from infusorial earth (Whipple's silica standard) which has heretofore been employed. The new standard may be thus defined "the standard of turbidity shall be a water which contains 100 parts of precipitated fuller's earth per million in such a state of fineness¹ that a bright platinum wire 1 mm. in diameter can just be seen when the center of the wire is 100 mm. below the surface of the water and the

¹ By grinding in an agate mortar and sifting through a No. 220 mesh sieve.

eye of the observer is 1.2 meters above the wire, the observation being made in the middle of the day, in the open air but not in sunlight, and in a vessel so large that the sides do not shut out the light so as to influence the results. The turbidity of such a water shall be 100."

Having obtained a fuller's earth fulfilling the above requirements, a stock suspension is made which contains 1 gm. of the dried earth in 1 liter of water. This is then tested, after diluting one part with 9 of water, with a 1 mm. platinum wire to ascertain whether it conforms with the above definition of turbidity 100. It may be slightly adjusted by adding a very little more fuller's earth or by adding more water. Should there be a marked discrepancy between the wire observation and the earth weighed out, a new standard should be prepared.

"Standards for comparison shall be prepared from the stock suspension by diluting with distilled water."

The following dilutions are suggested, 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, 90, and 100.

Turbidities below 20 are best compared by placing the sample side by side with the standard in bottles of clear white glass of the same size and shape, the observation being made sideways through the bottles toward the light, noting the appearance of some object, such as the division of the windows or of heavy black lines ruled on white paper. When the outlines of the objects observed through the bottles are seen with the same distinctness through the sample and standard, the two have equal turbidities.

Turbidities of from 20 to 100 are best compared by using 100 cc. Nessler tubes and looking through *sideways* with ruled paper as the means of measuring the change in distinctness of outline.

Samples having turbidities of over 100 should be diluted with known amounts of distilled water.

Before the standard suspensions and samples may be compared each must be shaken thoroughly enough to break up all agglutinated masses and insure a complete and uniform suspension of the fine particles.

For field work a turbidity rod with a 1 mm. platinum wire should be employed. A standard turbidity rod properly graduated may be purchased from the manufacturers.¹ Or if it is necessary or desirable that it shall be "home-made" the distance graduations may be obtained by applying to the U. S. Geological Survey for the Circular relating to "Measurement of Turbidity and Color."²

For the determination in the laboratory of turbidities over 100, Jackson's Candle Turbidimeter may be employed. "This consists of a graduated glass tube with a flat polished bottom, enclosed in a metal case. The graduated tube is held over an English standard candle and so arranged that one may look vertically down through the tube and see the image of the candle. The observation is made by pouring the sample of water into the tube until the image of the candle just disappears from view."

The graduations on the tube of the instrument correspond to silica standard turbidities.

Observations must be made in a darkened room or in a blackened box with a dark cloth over the head.

Precautions. Always see that the spring below the candle is free, so that it shall maintain the candle pushed up in the socket as far as it will go.

Never place the empty tube over the flame and start pouring in the sample. Always pour a little of the sample into the tube before holding it over the flame.

Never try to make an observation if any material or soot has collected on the bottom of the tube and carefully avoid spilling any sample in pouring into the tube, thus

¹ Builder's Iron Foundry, Providence, R. I.

² Or see Jour. Infect. Dis., May Supplement 1905, page 18.

preventing any material from falling down the sides and coating the bottom.

It is necessary to work with great rapidity in order that satisfactory results may be obtained.

All turbidity observations are to be recorded and reported in whole numbers corresponding to parts per million suspended fuller's earth, as follows.¹

Turbidity between 1 and 50 to nearest unit.

51 and 100 to nearest 5.

101 and 500 to nearest 10.

501 and 1000 to nearest 50.

1001 and above to nearest 100.

THE SANITARY CHEMICAL ANALYSIS OF A SAMPLE OF WATER.

In order that the sample of water shall not change in composition while an examination is being conducted, it is essential that the determinations of the required constituents be begun as soon as practicable after the taking of the sample, and pushed to completion as rapidly as possible. It being of course understood that the sample must be kept cool until the work is finished.

In order that this may be properly accomplished, the following outline is suggested as a guide for the beginner.

A. Start at once the purification of the apparatus for determinations of ammonia. Have this completed, if possible, just before the sample arrives.

B. Note the appearance of the sample; the amount and character of the sediment and suspended matter.

1. Fill pipette for determination of dissolved oxygen.

¹ U. S. Geological Survey System.

2. Determine free CO_2 .
3. Shake the bottle and observe the odor; pour out a portion and note odor cold and hot.
4. Start determination of nitrogen as Free and as Albuminoid Ammonia.
5. Determine color and turbidity while ammonias are distilling.
6. Start determination of nitrogen as nitrites.
7. Start determination of oxygen consumed.
8. While 7 is in progress determine alkalinity (or acidity).
9. Determine chlorine.
10. Start determination of nitrates; after evaporation, the dry residue may stand until a convenient opportunity to complete the determination.
11. Determine carbon dioxide as bicarbonates.
12. Determine mineral acid hardness.
13. Determine total solid residue, loss on ignition, solids in suspension.
14. Determine PO_4 .
15. Determine SO_4 .
16. Determine iron, manganese, etc.
17. Make such other special tests as may be necessary and start upon the mineral analysis (Boiler Water Analysis).

The determinations covered in the outline down to No. 13 must be completed in the first period of laboratory work

BACTERIOLOGICAL EXAMINATION OF WATER.

This branch of Sanitary Water Examinations is at once the most important and the least satisfactory. It is the most important since in the light of our present knowledge no water examination can be considered as yielding the information upon which to base a really sound opinion as to the potability of the sample analyzed without evidence as to the nature of the bacteria present, and further because in the control of filtration plants the resulting bacterial examination affords practically the only criterion of the efficiency of purification, and the least satisfactory for the reason that our knowledge of the bacteria found in water is at present very meagre and our methods crude, difficult of application, unreliable—within certain limits,—and not giving the necessary data in a short enough time.

American chemists are at present quite generally agreed upon the methods for the chemical analysis, but there is still much difference of opinion as to the best methods to adopt for the bacteriological examination.

A most serious difficulty is the impossibility of obtaining culture media of sufficiently constant composition to yield uniform results. Space does not permit a discussion of the problems involved, moreover, this matter is treated at length in the lecture course on Potable Water.

PREPARATION OF CULTURE MEDIA.

The directions given below for the preparation of culture media for the bacteriological examination of water are in the main not in accord with the standard methods of the American Public Health Association, the authors having found it impracticable to closely adhere to the standard

methods in the preparation of media on a large scale, and believe that in a number of instances, the standard methods do not yield media as satisfactory as those made as described below, at least so far as the waters of Central New York are concerned.

The directions given are to be considered as provisional and subject to change. In view of the results now being obtained by the authors in the study of culture media for water analysis there is every reason to believe that the directions as printed here will be subject to revision. All necessary changes will be posted on the blackboard or bulletin board.

Before proceeding to the preparation of media, see that all the enamelled steel ware, pails, kettles, etc., are scrupulously clean.

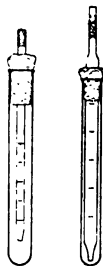
Wash in hot water and dry all glassware so that it is free from spots of grease or dirt.

Place the Petri dishes in a dry air sterilizer and heat to 140° to 160°C. for two hours or more. Allow to cool slowly in the sterilizer.

Clean and dry the 100 cc. bottles used for sample taking. See that the stoppers fit properly, cover with tin foil, pressing the foil closely about the neck of the bottle, tie a piece of clean muslin over the top and sterilize at 140°C. to 160°C. for two hours. Allow to cool slowly in the sterilizer.

Allow the water pipettes to stand in a cylinder in cleaning mixture over night, rinse thoroughly until *every trace* of acid has been removed using filtered distilled water in the rinsing. Dry thoroughly. Insert a *loose* cotton plug in the upper end of the pipette and wind a closely fitted but not too compact ring of cotton about 2 cm. wide around the middle of the pipette, making this ring of such size as to just nicely fit into a 15 cm. test tube for a distance of about

1 cm. Fit each pipette into a tube in this manner, pushing the pipette down into the tube as far as it will go, being careful to always have at least 1 cm. of the cotton ring projecting outside the test tube. See Fig 2. Sterilize in a dry air sterilizer at 140°C . to 160°C . for two hours and allow to cool down in the sterilizer.

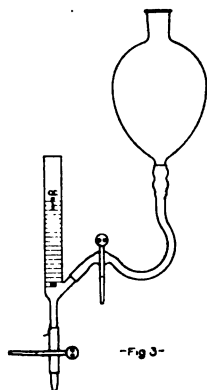


-Fig 2-

All apparatus used for bacterial cultures must be exceptionally well cleaned and wiped dry. Usually a treatment with cleaning mixture is essential to remove all traces of grease from glassware.

Caution. Never attempt to clean any apparatus which has contained cultures from water examinations until the apparatus and contents have been thoroughly heated in the autoclave at 120°C . for ten minutes or for one hour in the Arnold sterilizers in steaming steam.

If through an accident drops of culture media containing living organisms are spilled on the work tables, floors or hands, pour on a few cubic centimeters of the formaldehyde disinfectant found on the reagent shelves and then clean up very thoroughly. Should any wounds result from breaking glass, etc., report at once to some member of the staff of instruction for advice as to the proper sterilization of the wound.



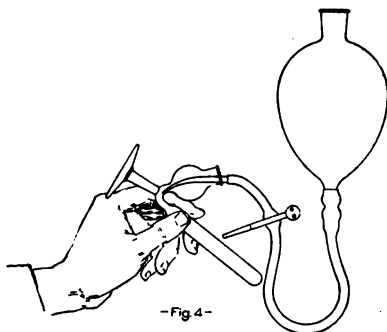
-Fig 3-

Carelessness in handling cultures will not be tolerated.

SPECIAL APPARATUS AND METHODS.

For the distribution of culture media in test tubes and flasks the measuring device shown in Fig. 3 will be found convenient and expeditious.

In filling fermentation tubes, hold the tube, bulb up, with the closed arm inclined at an angle of about 30° , and introduce the medium by allowing it to flow in from a slightly curved tube attached to a reservoir by a rubber



tube provided with a pinchcock, see Fig. 4. Never fill the bulb of a fermentation tube more than one-quarter full.

STERILE WATER.

Boil a liter of tap water until all the iron and as much as possible of the calcium and magnesium carbonates are precipitated. Filter. Distribute in wide short test tubes, measuring 9 cc. into each tube. Plug. Place in the autoclave and raise the pressure as rapidly as possible to one-half atmosphere, shut off the gas at once, and as soon as the tubes can be removed immerse them in cold water.

DEXTROSE-PEPTONE.

Aqueous solution containing 2 per cent. pure dry dextrose, 3 per cent. Witte peptone and 0.5 per cent. potassium chloride. It is usually unnecessary to adjust the reaction of the medium when using University tap water since the tubed medium will have a reaction of between $+1.5$ and $+1$. per cent.¹ Measure out the proper volume

¹ By a reaction of $+1\%$ is meant an acidity toward phenolphthalein equivalent to 10 cc. of normal HCl per liter. N. NaOH and N. HCl are used in adjusting reactions.

of tap water, heat to boiling (having just weighed the container and then container and water) boil briskly for about three minutes, turn the gas stove very low, add the peptone, stirring until practically all is dissolved, then add the dextrose, raise the gas flame until the liquid comes to a brisk boil, filter; if not clear, pour through the filter again. Let stand for several days in the ice chest and filter again.

The final filtrate should be perfectly clear and have a light straw color.

Fill a series of 15 cm. test tubes measuring 9 cc. into each tube, plug with absorbent cotton, sterilize in the autoclave, cool rapidly by immersing in cold water the wire basket containing the sterilized tubes.

Also fill a series of fermentation tubes, plug loosely with absorbent cotton, sterilize in the autoclave and set aside for use.

LACTOSE-PEPTONE.

Dissolve in boiled tap water 3 per cent. of Witte peptone, 2 per cent. of lactose and 0.5 per cent. of potassium chloride. Prepare exactly as directed under dextrose-peptone, filter, distribute in fermentation tubes, plug with cotton, sterilize in the autoclave and set aside for use.

LACTOSE-PEPTONE NEUTRAL RED. (Stokes' medium)

To the lactose peptone solution described above, add neutral red in the proportion of 0.1 gram per liter. Adjust the reaction so as to have it about + 1.5 per cent. Filter. Distribute in fermentation tubes, sterilize in the autoclave as usual and set aside for use. (Stokes, J. Infect. Dis., 1, 341 (1904))

B. coli and closely related varieties give in this medium in forty-eight hours a greenish yellow fluorescence in the closed arm of the tube, a port wine color in the bulb with a sharp line of demarkation between these colors, and 30 to

50 per cent. of gas with a gas ratio $H:CO_2$ = approximately 2:1.

LACTOSE-LITMUS BILE OR LACTOSE-LITMUS BILE SALT.

Prepare a lactose-peptone solution as previously described, add 0.5 per cent. "sodium taurocholate," boil for a few minutes and filter until a clear filtrate is obtained, then add a boiled and filtered solution of purified litmus, drop by drop, until colored distinctly blue. Fill a series of fermentation tubes, sterilize in the autoclave at one-half atmosphere, cool and set aside for use.

Note. The "sodium taurocholate" of commerce consists almost wholly of sodium glycocholate. These two compounds are believed to have substantially the same inhibiting action toward water bacteria.

GELATINE.

Prepare a meat broth by boiling finely chopped lean and perfectly fresh beef with tap water in the proportion of 500 grams of flesh to one liter of water, until the flesh is very thoroughly cooked. This will require boiling for from one to five hours depending upon the amount of material being used. The loss of water through evaporation must be replaced, from time to time. Strain through cheese cloth, squeeze in a meat press, and set the liquid aside to cool, when cold and the fat has hardened, filter through a wet folded filter. Weigh the resulting broth and add sufficient "gold label" gelatine to make a 10 per cent. solution (after the gelatine is added) and sufficient Witte peptone to give a peptone content equal to 2 per cent. Warm in an autoclave, with the cover off, or in a water bath with constant stirring and with care that the temperature does not rise over 50°C. When all has dissolved, carefully add normal soda solution until just neutral to *neutral* litmus, mix very thoroughly, add the whites of one or more eggs according

to the quantity of the medium. Place the pail containing the material in an autoclave, containing sufficient water to wet the bottom of the container, heat with the cover on but not screwed down until the temperature of the liquid has reached 85° to 90°C. Remove, strain through double cheese cloth or through a thin layer of absorbent cotton or cotton flannel. Weigh the strained liquid, remove an aliquot part (50 to 100 grams) and determine its acidity using phenolphthalein as indicator and normal sodium hydroxide. Adjust the reaction so that it will have an acidity to phenolphthalein equivalent to 10 cc. normal HCl per L. Mix very thoroughly, place in the autoclave and rapidly run up the pressure to 1 atmosphere, shut off the gas, allow the pressure to fall to zero, open, remove the gelatine and filter at once through a wet, folded, webbed filter supported on a layer of absorbent cotton in the funnel, the filter and funnel having first been thoroughly heated by pouring boiling water through.

The perfectly clear, almost colorless gelatine is measured into 15 cm. test tubes while still hot, 8 cc. being added to each tube with care that no drops of gelatine are allowed to fall upon the upper two centimeters of the tube. The tubes are then plugged with absorbent cotton plugs, placed in wire baskets, and again placed in the autoclave. The temperature is rapidly raised to one-half atmosphere and the gas shut off at once. As soon as the pressure reaches zero, the baskets of tubes are removed, allowed to stand for about five minutes, then set in pans of cold water to cool. When cold, the tubes are transferred to an ice chest for preservation for use.

AGAR-AGAR.

Prepare meat broth in the manner described under Gelatine, but using for its preparation a bulk of water equal to only two-thirds of the final volume of the medium.

While this is being prepared, soak the proper weight of shred agar-agar (20 gms. per L.) in water equal to the remaining one-third. Weigh the dish and contents. Allow to stand over night in a cool place. In the morning pour off the water from the agar, squeeze out the excess of moisture from the swollen shreds, place the dish and contents on a balance and add water until the original weight is again obtained. Place the dish on a water bath or on a tripod over a Bunsen burner and heat until the agar is dissolved, replacing the water lost by evaporation. Heat the meat broth after filtering off the fat, add 2 per cent. of Witte peptone, then the melted agar, stir. Now add normal sodium hydroxide solution until the reaction is neutral to *neutral* litmus paper. Cool to 50°C., add the white of one egg, stir very thoroughly, place in the autoclave until the albumen is completely coagulated, strain through cheese cloth, adjust the reaction to +1.0 per cent., heat in the closed autoclave until the temperature is that of boiling water. Filter at once while very hot through a hot wet webbed filter. Distribute the filtrate in 15 cm. plugged test tubes, running 8 cc. into each tube. Place in the autoclave, close and run up the pressure to one-half atmosphere, shut off the gas at once, and as soon as the pressure has fallen to zero, open, and remove the tubes. After cooling for about five minutes place the basket and tubes in cold water until partly cooled, remove, allow to drain and when the medium has hardened, set aside in a cool place for use.

LACTOSE-LITMUS AGAR.

Prepare agar-agar exactly as above adding, just before the adjusting of the reaction one per cent. of lactose.

To the final filtrate add just enough of a boiled and filtered solution of litmus to color the medium a distinct bluish violet. Distribute in tubes and sterilize as usual.

A better plan for preparing the lactose litmus media

consists in adding from 3 to 5 drops of sterile litmus solution to the lactose peptone solution after the water sample has been added and in the case of lactose agar adding the same amount of sterile litmus solution to the warm agar just before pouring into the Petri dish containing the 1 cc. of water.

Note. Instead of lactose-litmus-agar many analysts use lactose-litmus bile-salt agar, it being much easier to recognize *B. coli* group organisms since the growth of many other acid forming organisms is inhibited. On the other hand it must not be overlooked that attenuated varieties of *B. coli* will not develop on this medium and further that such attenuated varieties are the rule rather than the exception during the winter months.

ESCULIN BILE-SALT PEPTONE SOLUTION.

Dissolve in a liter of distilled water 20 grams of Witte peptone, 0.5 gram of esculin (Merck) and 0.5 gram of citrate of iron. Test the reaction. It should be $+0.6$; if not, adjust so as to obtain such an acidity. Filter and add 3 grams "sodium taurocholate." Distribute in 15 cm. plugged test tubes, using 10 cc. for each tube.¹

This medium turns black when fecal organisms (and several other species) are present. The development of a black color is in the opinion of the writers not proof positive of the presence of *B. coli* since there appear to be a very few other species of bacteria (soil bacteria?) and at least two species of mould (*Aspergillus*?) found in our local waters which also give rise to the formation of a black color in the tubes and to black colonies on esculin bile-salt agar. The liquid medium appears to be much more favorable to the growth of *B. coli* forms than the solid and is therefore a very valuable presumptive test. Attenuated varieties of *B. coli* appear to be more easily found by esculin bile-salt

¹ Harrison and Vanderleek, Cent. f. Bak 51, 607, (1909).

peptone solutions than by either lactose neutral red or lactose litmus bile-salt.

ESCULIN BILE-SALT AGAR.¹

“Boil until dissolved 15 grams of agar, 2.5 grams of commercial bile salt, and 10 grams of peptone (Witte) in 1000 cc. of distilled water. Neutralize, with a normal solution of sodium hydrate. Cool below 60°C., add the whites of two eggs, bring to the boil and filter as soon as the albumen has coagulated properly. Try the acidity and neutralize if necessary and then add to the clear hot filtrate 1 gram of esculin (Merck) and 1 gram iron citrate scales (Merck). After these substances are dissolved, test the acidity with decinormal soda solution. It will be found to be about + 0.6. In case the acidity is too high add alkali until the reaction is + 0.6.”

MEDIUM FOR TESTING FOR HYDROGEN SULPHIDE PRODUCTION.

The concentration of the medium after the addition of the water sample should be peptone 3 per cent.; potassium chloride, 0.5 per cent.; reaction, + 1.0 to + 1.5 per cent.

At present it is the usual custom to perform the test on a volume of 90 cc. to 100 cc. of the sample. With this end in view prepare a solution containing Witte peptone, 30 per cent, potassium chloride 5 per cent, reaction unadjusted. Filter. Distribute in 100 cc. flasks with a mark on the neck for 100 cc., placing 10 cc. of the medium in each flask. Plug. Sterilize in the autoclave at one-half atmosphere as usual. (Method of Chamot and Redfield.)

MEDIUM FOR PRESUMPTIVE TESTS FOR FECAL ORGANISMS.

This medium is made in order that a large volume (90 to 100 cc.) of the sample may be employed for the detection of the presence of members of the colon group of organisms.

Prepare a 30 per cent. solution of Witte peptone, add

¹ Harrison and Vanderleek, Cent. f. Bak., 51, 607 (1909).

5 per cent. of potassium chloride, 20 per cent. dextrose and 2 per cent. of crystallized phenol. Filter and keep in a capped and plugged culture flask in a cold place. Sterilization by heat is unnecessary since the quantity of phenol present is sufficient to sterilize the medium.

STEPS IN THE BACTERIOLOGICAL EXAMINATION OF WATER SAMPLES

Since most waters examined by the analyst contain large numbers of bacteria per cubic centimeter and counts of the number present becomes impossible when 1000 or over are present per cubic centimeter, and are very inaccurate should the number of colonies on a plate be greater than 150 to 250, it is essential that the sample be so diluted with a known volume of sterilized water as to bring the colony counts within the limits just given. With this end in view first make the necessary dilutions as described below.

Take a tube containing 9 cc. of sterilized water, flame the cotton plug, loosen it in the tube and flame again. Set this tube in a test tube rack or small beaker. Shake the bottle containing the water sample twenty-five times, loosen the stopper. Take up a sterile pipette in its tube in the left hand and thoroughly flame the cotton ring, pull out the pipette with the right hand and lay down the protective tube, remove the stopper from the sample bottle, insert the pipette and draw up 1 cc. of the sample and stopper the sample bottle at once, the stopper having been held in the hand. With the left hand now remove the cotton plug from the sterile water tube, run in one cc. of the sample, reinsert the cotton plug which has been carefully held in the fingers in the meantime. Lay aside the pipette (or proceed to the inoculation of media with the undiluted sample.) Care-

fully shake the tube from side to side about twenty-five times, avoiding any splashing on the cotton plug. Portions of one cubic centimeter (equal to 0.1 cc. of the original sample) are distributed in the proper media and Petri dishes, using a clean sterile 1 cc. pipette. One cubic centimeter from this tube is also introduced into a second tube of sterile water, mixed as before, and one cubic centimeter portions are taken therefrom and distributed in plates and tubes, each cubic centimeter representing 0.01 cc. of the original sample.

Whenever it is necessary to employ less than one cubic centimeter of a sample of water for analysis the above dilution method must be employed. Never try to distribute 0.1 and 0.01 cc., using finely graduated pipettes, it being essential that we work with media always with as nearly as possible similar concentrations.

Great care must be exercised by the beginner to avoid touching with the fingers the lower portions of the stopper of the sample bottle, those of the cotton plugs of the water tubes, and that part of the water pipette below the cotton ring. The beginner must also learn to work very rapidly so as to avoid the introduction of bacteria from dust blown or falling into apparatus and media being used.

Into a series of clean sterilized Petri dishes introduce 1 cc. of the well shaken sample. Into another series 1 cc. of the first dilution with sterile water = 0.1 cc., and into another series 1 cc. of the second dilution with sterile water = 0.01 cc. The water is introduced into the dishes by first filling a sterile water pipette with the sample, holding it in the right hand, raising the cover of a dish on one side, only just high enough to permit the insertion of the pipette, allowing the 1 cc. of sample to flow out and closing the dish. Work carefully but very rapidly. Never uncover the Petri dish completely.

Melt in the water bath tubes of gelatine, agar, etc.

When melted, cool to 40°C. Then take them out one at a time, hold them almost horizontally in the right hand, flame the cotton plug, remove it with the left hand and drop it on the table, flame the open end of the tube, raise the cover of a Petri dish just high enough to permit introducing the tube under the cover and empty it, withdraw the tube, lay it down and lower the cover. Take up the plate with both hands and mix the medium with the sample by carefully tilting the dish back and forth, giving at the same time, the liquid contained a slightly rotary motion until a uniform mixture results. Great care must be exercised in tilting the dish that the liquid does not run over the edge. But little practice is required to enable the analyst to work very rapidly and accurately. *Never* inoculate gelatine, agar, etc., in the tubes and then pour into the dishes.

As soon as the agar in the agar plates has set, turn them bottom side up. Agar in setting always squeezes out little drops of water like dew drops, these, if the plates are allowed to remain in the position as plated, flow together in spots, thus leading to overgrowths. Moreover, were the agar plates placed in an incubator at 38°C. in the normal position the water in the culture medium would be vaporized and the vapor rising would condense upon the cover. Thus there would be two sources of trouble, first the agar would rapidly dry and second, the condensed moisture forming large drops would fall back upon the surface of the culture medium and cause overgrowths. Covers of porous earthenware have been proposed to prevent overgrowths, but the authors have always found them to be unsatisfactory. Turning the agar plates upside down has been the practice in this laboratory for some fifteen years or more.

Test tubes and fermentation tubes of media are best inoculated by holding them inclined in the left hand, flaming and loosening the plug, then with the pipette grasped in the thumb and second finger of the right hand

the cotton plug of the tube is removed by the fourth and little fingers of the right hand, keeping that part of the plug that is to go back into the tube away from the hand and also keeping it from touching any object as desk top, bottles, etc., the pipette is then emptied into the tube, and the cotton plug replaced. The worker may adopt, however, such a method as will be the least awkward and at the same time permit of the most rapid manipulation.

After inoculating the test tubes, shake from side to side, until very thoroughly mixed. In the case of fermentation tubes, shake from side to side with a rotary movement until the contents of the bulb are uniformly mixed, then tip the tube until a few bubbles of air rise in the closed arm, then tip the other way expelling all the air, repeating several times.

THE CHOICE OF MEDIA, AND NUMBER OF CULTURES.

This will necessarily depend upon the character of the water and the nature of the data wanted.

If dealing with a water concerning which there is the least suspicion it is safe to make no less than the inoculations listed below. Specific directions will be posted upon the blackboard in the laboratory for each water sample to be examined.

Gelatine plates at 20°C. in air. At least two with 1 cc. each; two with 0.1 cc. each and if believed to be very bad, two with 0.01 cc. each.

Agar plates at 20°C. At least two with 1 cc. each, two with 0.1 cc. each.

Agar plates at 38°C. Two with 1 cc. each; two with 0.1 cc. each.

(Agar Lactose Litmus, at 38°C. Two with 1 cc. each; two with 0.1 cc. each.)

(Agar Esculin Bile Salt at 38°C. Two with 1 cc. each; one with 10 cc. in large dish.)

Fermentation Tubes.

Dextrose-Peptone; ten with 1 cc. each; five with 0.1 cc. each.

(Lactose Peptone: ten with 1 cc. each; five with 0.1 cc. each.)

(Lactose Litmus Bile Salt: ten with 1 cc. each; five with 2 cc. each.)

(Lactose Neutral Red: ten with 1 cc. each; five with 2 cc. each.)

Dextrose-Peptone to which five drops of 5 per cent. phenol are added.¹ Ten with 1 cc. each, five with 2 cc. each. Incubate at 42°C.

(Esculin Bile Salt: ten with 1 cc.; five with 0.1 cc. and five with 2 cc. Incubate at 38°C.)

Special Cultures.

a. 90 cc. sample added to 10 cc. of medium for H₂S Formation Test. Incubate at 38°C.

b. To 90 cc. sample in sample bottle, add 10 cc. presumptive test medium (phenolized-dextrose peptone) incubate at 38°C.

c. To 10 cc. sample in a sterile test tube, add 1 cc. of presumptive test medium, incubate at 38°C.

In the list above the media bracketed are rarely all used on one and the same water sample. Usually one or possibly two are selected according to the preference of the analyst. The beginner is strongly urged, however, to run the entire series on a number of water samples until he becomes somewhat familiar with the good points and the failings of each kind of culture media.

When dealing with fermentation tubes carefully record in the note book, the appearance of the culture at the end of 24, 48 and 72 hours, whether gas is formed and if so, how

¹ Method of Vincent, (*Macé Traité de Bacteriologie*).

much. Mark the height of the gas on the closed arm by means of a "glass pencil." When making the final examination, determine the H to CO_2 ratio by filling the bulb with 1:1 potassium hydroxide, shake from side to side to mix, then incline the tube backwards to cause the flow of the alkali into the closed arm. Repeat the process several times until the liquid ceases to rise in the arm—mark the height, add more potash and mix again. As soon as all the CO_2 is absorbed find the gas ratio.

The colonies on agar plates incubated at 37° to 38°C . should be counted at the end of twenty-four hours, and again after forty-eight hours.

The colonies on gelatine and agar plates at 20°C . should be counted after forty-eight hours' incubation and if possible again after seventy-two hours. When the number of colonies developing upon a plate are few it is the best plan to allow the plates to remain as long as possible in the incubator before making the final counts in order that slow growing species may become apparent and that all the species present may develop sufficiently to exhibit their characteristic appearances.

The following table compiled from data collected by Stokes in his study of 567 fermentative water bacteria¹ is offered as an aid in the interpretation of fermentative phenomena which may be observed by the student in the fermentation tubes inoculated with water samples. It must be understood that the values for gas formed and gas ratios are typical average results and that variations will surely be met with.

¹ See also Jordan, *Journ. of Hyg.*, 3, 1 (1903).

TYPES OF FERMENTATIVE BACTERIA IN WATER.¹

Group Typical Organism	Behavior on Gelatin	Gas Formation in Forty-eight Hours and Gas Ratio CO ₂ : H				Neutral-red Lactose yellow red contrast	In Lactose Neu- tral-Red up to 50% gas CO ₂ : H = 1:2 and yellow red contrast reaction
		Dextrose	Lactose	Sucrose	Sucrose		
I. <i>B. vulgaris</i>	Liquefying	20%	1:2 6 to 10%	25%	1:2	Positive	Negative
II. <i>B. cloacae</i>	Liquefying	60 to 100%	2:1 0 to 10%	60 to 100%	2:1	Positive or Negative	Negative
III. <i>B. cloacae</i>	Non-liquefying	60 to 100%	2:1 0 to 10%	60 to 100%	2:1	Negative	Negative
IV. <i>B. gasoformans</i>	Liquefying	80%	2:1 90 to 100%	90%	2:1	Negative	Negative
V. <i>B. gasoformans</i>	Non-liquefying	80%	2:1 90 to 100%	90%	2:1	Negative	Negative
VI. <i>B. enteritidis</i>	Non-liquefying	40 to 50%	1:2 0	0	2:1	Positive	Negative
VII. <i>B. prodigiosus</i>	Liquefying	40%	1:2 90%	2:1 80%	2:1	Negative	Negative
VIII. <i>B. pyocyaneus</i>	Liquefying	Usually no fermentation of sugars				Negative	Negative
IX. <i>B. pseudo coli</i>	Non-liquefying	85%	1:2 40 to 70%	90%	2:1	Negative	Negative
X. <i>B. coli</i>	Non-liquefying	40 to 50%	1:2 40 to 50%	0 to 30%	1:2	Positive	Positive

¹ Arranged from Tables of W. G. Stokes, J. Infect. Dis., 1, 341 (1904).

ESTIMATION OF PUTRESCIBILITY IN SEWAGE EFFLUENTS.

Method of Laboratory Section, Am. Pub. Health Association.

The Committee on the revision of standard methods for the chemical analysis of water and sewage have recommended the following:¹

“Samples should be collected in glass-stoppered bottles of 150 or 200 cubic centimeters capacity. No special precautions are necessary in collecting samples of ordinarily good effluents that are fairly high in dissolved oxygen. If the dissolved oxygen is low, precautions similar to those used in collecting dissolved oxygen samples should be observed. A one-tenth per cent. solution of methylene blue, preferably Merck's double zinc salt, is used as indicator. One-half cubic centimeter of this solution is added to each of the samples, which are then incubated, preferably at 20°C., for four days, and observations are made at least once a day. If quick results are desirable the incubation may be made at 37°C. by adopting suitable precautions to prevent the loss of dissolved oxygen. For this purpose the sample is taken in a bottle fitted with a rubber stopper through which an ordinary medicine dropper is introduced by half its length. Before inserting the stopper into the filled bottle the bulb of the medicine stopper is compressed. As the solution warms up this bulb takes care of the additional volume necessary and prevents the loss of dissolved oxygen. The samples in which the methylene blue becomes decolorized are recorded as having a relative stability corresponding to the time required for reduction, as given below. Those that are blue at the end of four days are given a relative stability value of 95. For more accurate work a longer period of incubation is recommended.

“The following table gives the relation between the

¹ Amer. J. Public Hygiene, 20 (1910), 367-370.

time of reduction at both 20° and 37° and the relative stability number.

RELATIVE STABILITY NUMBERS.

t20	t37	s	t20	t37	s
0.5	...	11	8.0	4.0	84
1.0	0.5	21	9.0	4.5	87
1.5	...	30	10.0	5.0	90
2.0	1.0	37	11.0	5.5	92
2.5	...	44	12.0	6.0	94
3.0	1.5	50	13.0	6.5	95
4.0	2.0	60	14.0	7.0	96
5.0	2.5	68	16.0	8.0	97
6.0	3.0	75	18.0	9.0	98
7.0	3.5	80	20.0	10.0	99

s—Relative stability or ratio of available oxygen to oxygen required for equilibrium. Expressed in per cent.

t20—Time in days to decolorize methylene blue at 20°C.

t37—Time to decolorize at 37°C.

Theoretical relation

$$s = 100 (1 - 0.794^{t_{20}})$$

$$= 100 (1 - 0.630^{t_{37}})$$

“A relative stability of 75 per cent. means that the effluent in question contains a supply of available oxygen equal to 75 per cent. of the amount of oxygen which the effluent will eventually require before it will have become perfectly stable. The amount of this available oxygen is estimated fairly well by the chemical determination of dissolved oxygen and nitrates. . . . In general, effluents having a relative stability greater than 90 per cent. may be discharged into any stream without danger of their consuming any of the oxygen of the water, because effluents

of such high stability will retain oxygen indefinitely on exposure to the air."

MICROSCOPICAL EXAMINATION OF WATER.

Laboratory work in this field may be elected in Chemistry 72, Microscopical Examination of Foods, etc., and is therefore not here discussed.

MINERAL ANALYSIS OF WATER FOR BOILER AND OTHER INDUSTRIAL PURPOSES.

Determine as already described:

1. Total Solid Residue, page 53. Solids in suspension, page 54.
2. Loss of Solids on Ignition, page 54.
3. "Organic Matter," page 54.
4. Alkalinity (or Acidity) Mineral Acid Hardness, page 45, 47.
5. Nitrates, Nitrites, pages 38, 39.
6. Chlorine, page 44.
7. Phosphates, Sulphates, pages 40, 41.
8. Direct Ammonia, page 34.
9. Free, Bicarbonate and Normal Carbonate CO_2 , pages 51, 52.
10. Iron and Manganese, pages 42, 43, if present in appreciable amount.

RAPID METHOD OF ANALYSIS, with the object of ascertaining the character of the salts in solution.

If the water is turbid or contains much suspended matter, filter, adding a little aluminum cream, if necessary. Evaporate one liter of the filtrate to dryness on the water bath in a No. 7 porcelain evaporator (or better in a platinum dish). When dry, place the dish for about two hours in an air bath at 100° to 105°C . Cool and extract the dry residue repeatedly with small portions of ethyl alcohol of

Sp. Gr. 0.92. (Method of Kennicut.) Rub up the residue with about 100 cc. portions each of the alcohol using a porcelain pestle for the purpose, and filter each portion through a 12 cm. filter. After five extractions have been made, having allowed each volume of alcohol to remain for a time in contact with the residue, test for complete extraction by evaporating a sixth portion of alcohol which has been rubbed up very thoroughly with the water residue. When the alcohol no longer dissolves any salts, remove the flask containing the clear alcoholic filtrate, place the evaporator with the residue under the funnel, punch a hole through the filter and wash the contents of the filter into the dish, using distilled water. There is thus obtained a residue insoluble in alcohol and a clear alcoholic solution. These two portions are each subjected in turn to analysis following the methods adopted for the analysis of a limestone.

Analysis of the Residue Insoluble in Alcohol.

To the contents of the evaporator add sufficient dilute hydrochloric acid to decompose all carbonates, keeping the dish covered with a large beaker cover to avoid loss. Rinse off the cover, and evaporate on the water bath. Add to the residue thus obtained sufficient strong hydrochloric acid (1:1) to moisten it, and evaporate to dryness on a water bath IN THE HOOD. Place in an air bath at 100° to 105°C. for 1 hour, add 25 cc. of dilute hydrochloric acid and 75 cc. of water, heat to boiling and filter through a 9 cm. ashless filter into a 250 cc. measuring flask. Wash well with small volumes of hot water containing a little HCl, being careful that the filter is completely washed yet the volume does not exceed 250 cc. (It not infrequently happens that a water high in solids will require filtering into a 500 cc. measuring flask.) The filter is dried, ignited, and weighed = SiO_2 (together with impurities).

In an aliquot portion of the filtrate (50 cc.) determine

the SO_4 by precipitation with barium chloride and long boiling, or colorimetrically.

In another portion (50 cc.) determine the Fe, Al, Ca, Mg, as follows: To the solution (50 cc.) in a 200 cc. beaker add 2 drops of concentrated HNO_3 and boil. Then add a drop of methyl orange and then cautiously, NH_4OH , until just alkaline. Make acid again with HCl and finally make alkaline with NH_4OH , using not more than three or four drops of the reagent in excess. Heat to boiling, filter, place a clean beaker under the funnel, redissolve the precipitate in HCl and again precipitate as before with NH_4OH , filter, wash with water containing a little NH_4Cl , ignite and weigh = $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$. The filtrates and washings are united, a few cubic centimeters of NH_4Cl solution added, then $(\text{NH}_4)_2\text{C}_2\text{O}_4$ in slight excess. Heat to boiling; filter. Dissolve the precipitate in HCl and reprecipitate by adding NH_4OH , heat to boiling and set aside for three hours or more to settle. Filter, receiving the filtrate in the beaker containing the filtrate from the first precipitation. Wash well, using hot water containing about one-eighth its volume of NH_4OH . Dry the precipitate, ignite and weigh = CaO . Or dissolve the precipitated oxalate in sulphuric acid and titrate with permanganate.

The filtrate from the precipitation of the lime is concentrated if necessary and when cooled sufficient NH_4OH is added to make the solution alkaline, then add ammonium phosphate solution drop by drop with constant stirring until in slight excess. After standing for a few hours, filter through a filter previously moistened with dilute NH_4OH . Wash with water containing one-eighth of its volume of NH_4OH . Dry, ignite, and weigh = $\text{Mg}_2\text{P}_2\text{O}_7$.

The filtrate from the magnesium determination is evaporated to dryness, in a platinum dish, and gently ignited IN THE HOOD until all ammonia fumes are driven

off. Cool, moisten with concentrated HCl and evaporate to dryness IN THE HOOD on a water bath. Ignite gently to constant weight = $\text{NaCl} + \text{KCl}$. (This residue always contains some phosphates. Make a microchemical test for the presence of phosphates to learn whether a correction is necessary. If so, determine the amount of PO_4 colorimetrically and correct.)

Analysis of the Alcoholic Extract.

Evaporate to dryness. Heat to 100° to 150°C . Extract with water and filter into a 250 cc. measuring flask.

In aliquot portions determine the SO_4 , SiO_2 , Fe, Al, Ca, Mg, Na and K in the manner just described.

Make all determinations in duplicate.

From the data thus obtained it is possible to make computations with sufficient accuracy to meet the requirements of the usual industrial conditions.

The alcohol method of Kennicutt has been suggested as it permits a sufficiently accurate determination of the calcium sulphate present and yields definite information as to the nature of the salts of magnesium and calcium in the total solid residue.

Results of analyses are now generally reported in parts per million of the ions present, e.g. Ca, Mg, Na, K, (NH_4) , CO_3 , HCO_3 , SO_4 , Cl, NO_3 , NO_2 , PO_4 , H, Fe, Al, Si. In the case of boiler waters, amounts in grains per U. S. gallons of 231 cu. in. are also usually given and in these cases instead of the ions it is more customary to report SiO_2 , Fe_2O_3 and Al_2O_3 , etc.

There are still requests for reports stating the combinations or forms in which the constituents actually exist in a water and it is to meet this that the method described has been selected. In order to compute the "Hypothetical Combinations" or "Calculated Constituents," it is neces-

sary for us to make certain hypotheses which appear to be warranted by the facts: these are—

Any magnesium found in the residue insoluble in alcohol must be in the form of carbonate.

Any SO_4 found in the residue insoluble in alcohol is there as CaSO_4 , this compound being insoluble.

All the calcium found in the residue insoluble in alcohol is there as carbonate, sulphate or phosphate.

The magnesium found in the alcoholic solution is there as sulphate, chloride or nitrate.

The calcium found in the alcoholic solution is in the form of chloride or nitrate.

Any SO_4 in the alcoholic solution must be as salts of magnesium, sodium, potassium or ammonium.

We further assume that in all ordinary waters the SiO_2 is present in colloidal form and that the iron and the aluminum compounds are also colloidal. Hence these are reported as SiO_2 and $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$.

With the above hypotheses as a basis, it is thus possible to obtain a fairly acceptable conception as to the nature of the salts in solution by proceeding as follows:

Insoluble portion.

1. Calculate all the Mg found to MgCO_3 and to $\text{Mg}(\text{HCO}_3)_2$, if bicarbonates are present. Calculate the CaCO_3 equivalent of the latter and subtract this value from the total alkalinity. The difference is equal to the CaCO_3 in the insoluble portion. (Providing no alkali carbonates are present in the water and there is no hydroxide alkalinity.)
2. Calculate all the SO_4 to CaSO_4 .
3. Find the amount of CaO in the CaSO_4 and in the CaCO_3 just calculated as present. Add these two values. The sum should be substantially equal to the CaO found on analysis of the portion insoluble in

alcohol. Should there be a discrepancy it will be necessary to review all work and calculations. Should PO_4 be present the CaO found is usually greater than the sum of the two CaO values calculated.

Soluble in Alcohol.

1. Calculate the Mg found, first to MgSO_4 ; then any excess of Mg, over that required by the SO_4 ; first to MgCl_2 , next to $\text{Mg}(\text{NO}_3)_2$.
2. Calculate all the NO_3 found first to $\text{Ca}(\text{NO}_3)_2$, any excess to $\text{Mg}(\text{NO}_3)_2$ and then to NaNO_3 or KNO_3 .
3. Calculate the soluble lime first as $\text{Ca}(\text{NO}_3)_2$; combine any remaining Ca as CaCl_2 .
4. Calculate the SO_4 to MgSO_4 and any excess to Na_2SO_4 or K_2SO_4 (very rarely $(\text{NH}_4)_2\text{SO}_4$).
5. Calculate the N as NH_3 (direct ammonia) to NH_4Cl , (or $(\text{NH}_4)_2\text{CO}_3$).
6. Find the amount of Cl in the combinations calculated (NH_4Cl , MgCl_2 , CaCl_2). Subtract the sum from the Cl found on titrating a portion of the water with AgNO_3 in the usual manner. The difference gives the Cl combined with NaCl and KCl. Calculate this difference to NaCl.
7. If sufficient potassium has been found to be present by qualitative analysis (microchemical) to materially effect the results, it will be necessary to separate the K and Na. Usually the discrepancy due to calculating all the residual Cl to NaCl and ignoring the presence of K is so small as to have no real significance in industrial problems. But it should be stated on the report—"K and Na calculated as NaCl."

Finally find the sum of the SiO_2 , Fe_2O_3 + Al_2O_3 in both soluble and insoluble portions. Record this as "Colloidal Matter (Inorganic)".

Find the sum of all of the constituents thus computed and add thereto the "Organic Matter." The final sum should differ only very slightly from the "Solids in Solution."

The authors believe the Kennicott method, while open to much criticism, offers the simplest solution of computing combinations. The expert in microchemical analysis can usually identify with little trouble in water residues the following constituents— $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$; alkali chlorides; alkali sulphates; alkali nitrates; CaCO_3 ; alums. If the data thus obtained are compared with the computed salts it is rare that there is a disagreement.

It is, however, not out of place to again state that it is far better to report results in ionic form than in hypothetical combinations, since in one case we are dealing with facts, in the other with conjectures.

MINERAL ANALYSIS OF INDUSTRIAL WATERS WITHOUT REGARD TO THE NATURE OF THE SALTS IN SOLUTION.

Determine as already described—

Total Solid Residue. Suspended Matter.

Alkalinity or Acidity.

Bicarbonates, Carbonates, Free CO_2 .

Nitrates.

Phosphates.

Chlorine.

Evaporate to dryness on the water bath one liter or more of the sample, using a porcelain or platinum dish. Cautiously heat the dry residue (covered) until the organic matter is carbonized, carefully avoiding heating to redness. Cool, add (IN THE HOOD) sufficient concentrated hydrochloric acid to completely moisten the residue, keeping the dish covered with a beaker cover. Heat on the water bath for about half an hour. Again moisten the residue with concentrated acid, rub down the residue from the sides of

the dish and thoroughly break up all lumps. Evaporate to dryness on the water bath. Heat in an air bath to 100°C . for about two hours.

Determination of Silica.

Add about 3 cc. of concentrated hydrochloric acid and sufficient hot distilled water to dissolve all soluble salts, keeping the volume as low as possible. Heat almost to boiling and filter through a 9 cm. ashless filter into a measuring flask. Wash, dry and ignite the precipitate in a weighed platinum crucible, $= \text{SiO}_2$. Treat the crucible contents with $\text{HF} + \text{H}_2\text{SO}_4$ in the usual manner and correct the weight of SiO_2 for any occluded impurities. If a non-volatile residue is obtained dissolve it in a very little concentrated hydrochloric acid and add to the filtrate from the SiO_2 .

Determination of Iron and Aluminum.

To about one-fifth of the filtrate from silica add two or three drops of concentrated nitric acid and boil. Precipitate the iron and aluminum with ammonium hydroxide, wash, dry, ignite and weigh. In a separate portion determine the Fe colorimetrically, or if the volume of the sample is too small to permit this fuse the weighed $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ with HKSO_4 in a platinum crucible, cool, dissolve in sulphuric acid (1:3). Filter and add to the filtrate about 1 cc. of concentrated nitric acid, boil for five minutes, cool, dilute and determine the iron colorimetrically.

Determination of SO_4 .

To the filtrate from the precipitation of the Fe and Al by ammonia add a drop of methyl orange, then dilute hydrochloric acid until neutral and finally about 1 cc. in excess. Heat to boiling and add drop by drop a hot solution of barium chloride to slight excess, stirring thoroughly during the addition. Place on the water bath for two or

three hours or allow to stand over night. Filter, wash with hot water, dry, ignite and weigh.

Determination of Calcium.

To an aliquot part of the filtrate from SiO_2 add carefully ammonium hydroxide until the odor is just discernible, heat to boiling and add ammonium oxalate in sufficient amount to convert all the Ca and Mg present into oxalates. Allow to stand two or three hours, decant the clear solution through a Caldwell-Gooch filter transferring as little precipitate as possible to the filter. Dissolve the precipitate in dilute hydrochloric acid, dilute to 50 cc. and reprecipitate with ammonium hydroxide, digest on the water bath for a few minutes, and transfer the precipitated calcium oxalate to the Caldwell-Gooch filter and wash with as small a volume as possible of water containing one-eighth of its volume of ammonium hydroxide. Set the filtrate aside for the determination of magnesium.

Punch out the platinum disk, asbestos and precipitate of calcium oxalate into a 250 cc. Erlenmeyer flask and wash all precipitate adhering to the crucible walls into the flask. Add sulphuric acid (1:3 or 1:4) in excess, warm to 60° or $70^\circ\text{C}.$, and titrate with a standard permanganate solution.

Determination of Magnesium.

To the filtrate from calcium add a drop of methyl orange indicator and sufficient hydrochloric acid to make it very slightly acid and concentrate on the water bath to a volume less than 200 cc., cool and add an excess of sodium phosphate solution, mixing thoroughly. Add ammonium hydroxide in such an amount as not to have an excess of over 3 or 4 cc. Let stand several hours. Wash by decantation with water containing one-eighth of its volume of ammonium hydroxide using a filter previously moistened with this wash solution. Carefully avoid a large amount of wash water. Dry, ignite and weigh as $\text{Mg}_2\text{P}_2\text{O}_7$.

Determination of Sodium and Potassium.

The filtrate from the determination of SO_4 (or a separate portion precipitated with barium chloride) is evaporated to dryness. Dissolve the residue in water, add barium hydroxide in very slight excess and filter off and wash the precipitated magnesium hydroxide.

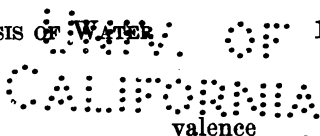
Heat the filtrate and add ammonium carbonate and a little ammonium hydroxide in sufficient amount to completely precipitate all barium and calcium, then add a few drops of ammonium oxalate. Allow to stand for a few hours, filter, wash and evaporate the filtrate to dryness on the water bath in a platinum dish. Heat IN THE HOOD gently below redness to remove ammonium salts. Cool. Dissolve in 5 or 10 cc. of water, warm gently and add a few drops of ammonium carbonate and ammonium hydroxide. Filter, wash, add a few drops of hydrochloric acid and evaporate to dryness on the water bath in a weighed platinum dish. Ignite gently to constant weight = $\text{NaCl} + \text{KCl}$. If more accurate results are wanted, dissolve the residue in water, filter, wash and ignite and weigh the filter. Correct the weight of the chlorides for any Ca or Mg compounds occluded.

APPLICATION OF ANALYSES TO BOILER WATER PROBLEMS, ETC.

The following formulas have been suggested by Herman Stabler¹ for the more common computations arising in industrial water problems where the results of analyses are expressed in ionic form. These formulas represent the greatest advance made in the interpretation of the results of the mineral analysis of water in recent years. As given below they have been copied with but little alteration in form or wording, with the permission of Mr. Stabler.

¹ Engineering News, 60, 355 (1908).

Symbols Employed.



r = reacting weight of radicle = $\frac{\text{molecular weight}}{\text{valence}}$

C_m = Colloidal Matter = $SiO_2 + Fe_2O_3 + Al_2O_3$.

S_m = Suspended Matter.

Symbols (Ca, Mg, Na, SO_4 , etc.) used in the formulas stand for parts per million of the radicles found on analysis.

"1. Coefficient of Corrosion.

$C = 1.008 (rH + rAl + rFe + rMg - rCO_3 - rHCO_3)$ or

$C = H + 0.1116 Al + 0.0361 Fe + 0.0828 Mg - 0.0336 CO_3 - 0.0165 HCO_3$.

Interpretation.

"A. If C be positive, the water will certainly corrode the boiler.

B. If $C + 0.0503 Ca$ (i.e. $1.008 rCa$) be negative, no corrosion will occur.

C. If C be negative, but $C + 0.0503 Ca$ be positive, corrosion may or may not occur."

2. *Scale Formation.* Sc = Pounds of Scale per 1000 gallons water.

$Sc = 0.00833 Sm + 0.00833 Cm + 0.3 rFe + 0.142 rAl + 0.168 rMg + 0.492 rCa$ or

$Sc = 0.00833 Sm + 0.00833 Cm + 0.0107 Fe + 0.0157 Al + 0.0138 Mg + 0.0246 Ca$.

In using this formula rCa must not be in excess of $(rCO_3 + rHCO_3 + rSO_4)$ or Ca should not exceed $(0.668 CO_3 + 0.328 HCO_3 + 0.417 SO_4)$.

The value Sc will correspond closely to the calculation of total incrusting matter calculated by the "Hypothetical Combination" method.

The character of the scale formed may be readily determined by the formula.

Quantity of Scale.

- a. Very little....Sc = not over 1.
- b. Little.....Sc = more than 1 but not over 2.
- c. Much.....Sc = more than 2 but not over 4.
- d. Very much...Sc = more than 4.

3. *Hard Scale.* Hs.

$$Hs = 0.00833 \text{ SiO}_2 + 0.0138 \text{ Mg} + (0.016 \text{ Cl} + 0.0118 \text{ SO}_4 - 0.0246 \text{ Na} - 0.0145 \text{ K}).$$

Where the value used for the parenthesis must not exceed 0.0118 SO₄ or 0.0283 Ca nor should it be less than zero.

Hs

- 4.
- Coefficient of Scale Hardness, h*
- =
- $\frac{Hs}{Sc}$
- .

Sc

Stabler suggests the following classification of Scale Hardness.

- a. Soft Scale, *h* = not more than 0.25.
- b. Medium Scale, *h* = more than 0.25, but not over 0.5.
- c. Hard Scale, *h* = over 0.5.

Foaming Coefficient.

- 5.
- $f = 2.7 \text{ Na} + 2. \text{ K}.$

$$\text{Run of boiler in hours, Rh} = \frac{\text{Capacity of boiler}}{\text{Consumption of feed water per hr. } f} \frac{k}{(-1)}$$

In which *k* is a constant = concentration of salts expressed in parts per million, causing excessive foaming. *k* varies with type of boiler.

Value of k according to Christie

Locomotive boiler	2,500 to 3,500
Stirling	4,000 to 5,000
Babcock & Wilcox,	
Heine, etc. (Water tube)	5,000 to 7,000
Horizontal return tubular boiler	8,000 to 10,000
Old style two-flue boiler	17,000

Classification of Waters.

- a. Non-foaming : f not greater than 60
- b. Semi-foaming : f greater than 60 but not over 200
- c. Foaming : f greater than 200.

Soap Consuming Power.

Whipple¹ finds that it requires approximately 201 pounds of the average ordinary soap to soften one million gallons of water for each part per million of hardness expressed as CaCO_3 .

$$6. S = 201 \times h.$$

where S = lbs. Soap per million gallons

h = Total Hardness.

$$201 \times h$$

$$7. \text{ Soap Cost. } cS = \frac{201 \times h}{20} = \text{Dollars per million}$$

gallons.

Stabler formula:

$$8. \text{ Soap Cost in cents per 1000 gallons} =$$

$$11 + 50.05 (r\text{Fe} + r\text{Al} + r\text{Ca} + r\text{Mg} + r\text{H}) \text{ or} =$$

$$11 + 1.79 \text{ Fe} + 5.54 \text{ Al} + 2.5 \text{ Ca} + 4.11 \text{ Mg} + 49.6 \text{ H.}$$

Water Softening Formulas—Stabler.

$$9. L = 0.26 (r\text{Fe} + r\text{Al} + r\text{Mg} + r\text{H} + r\text{HCO}_3 + 0.0454 \text{ CO}_2) = 0.00931 \text{ Fe} + 0.0288 \text{ Al} + 0.0213 \text{ Mg} + 0.258 \text{ H} + 0.00426 \text{ HCO}_3 + 0.0118 \text{ CO}_2.$$

$$10. Sd = 0.465 (r\text{Fe} + r\text{Al} + r\text{Ca} + r\text{Mg} + r\text{H} - r\text{CO}_3 - r\text{HCO}_3) = 0.0167 \text{ Fe} + 0.0515 \text{ Al} + 0.0232 \text{ Ca} + 0.0382 \text{ Mg} + 0.462 \text{ H} - 0.0155 \text{ CO}_3 - 0.00763 \text{ HCO}_3.$$

L = lbs. 90% CaO required per 1000 gallons.

Sd = lbs. 95% Soda Ash required per 1000 gallons.

The cost of softening with lime at 0.3 cents per pound and Soda Ash at 1.2 cents per lb. will be

¹ Whipple, "The Value of Pure Water," Wiley, N. Y., 1907, page 27.

11. Cost in cents per 1000 gallons.

$$\begin{aligned} &= 0.636 (r\text{Fe} + r\text{Al} + r\text{Mg} + r\text{H}) + 0.558 (r\text{Ca} - r\text{CO}_3) + 0.00354 \text{ CO}_2 - 0.48 r\text{HCO}_3 \text{ or} \\ &= 0.0228 + 0.0704 \text{ Al} + 0.0522 \text{ Mg} + 0.631 \text{ H} + 0.0279 \text{ Ca} + 0.00354 \text{ CO}_2 - 0.0186 \text{ CO}_3 - 0.00787 \text{ HCO}_3. \end{aligned}$$

Interpretation.

In formula (10) a negative value for Sd shows that no soda ash is required. Cost will then be (9) Lime \times 0.3.

In practice omit in formulas 9, 10 and 11, Fe, Al, and H unless the values actually found are sufficiently high to materially affect the computations.

EVALUATION OF "BLEACH."

Commercial "chloride of lime" or calcium hypochlorite is generally purchased on a guaranteed content of 35 per cent. "available chlorine" when employed in water sterilizing plants. (What is the theoretical content "available chlorine"?)

"Bleach" is theoretically $(\text{CaCl}_2 + \text{Ca}(\text{OCl}_2))$ but the commercial product contains these compounds in variable proportion and in addition some hydroxide and often some chlorate.

"Bleach" is placed on the market by the American manufacturer in 700 lb. drums and imported in 450 lb. barrels. It is repacked by dealers in 100 lb. drums, 10 lb. zinc cans and 1 lb. zinc cans and cartons. Since bleach is very unstable and rapidly deteriorates it is necessary that an analysis be made each time a solution is needed to fill the tanks of the sterilizing plant.

Drums or large cans should be sampled with a sampler constructed like a cheese trier, forced from top to bottom of the container, withdrawn, the bleach scraped out with a spatula into a dish and carefully sampled.

Five grams are introduced into a stoppered weighing

tube, carefully weighed, washed into a porcelain evaporator, rubbed up with a pestle with sufficient water to make a thin uniform paste and then completely transferred to a 500 cc. measuring flask. Shake the thoroughly mixed milky solution and measure out at once, (before any subsidence has taken place) portions of 20 cc. each for titration by method A or method B.

*Method A.*¹

Introduce the 20 cc. portion of the sample into a 250 cc. Erlenmeyer flask. Add from 15 to 20 cc. of a 10 per cent. potassium iodide solution, then acetic acid sufficient to make the solution acid, and 100 cc. distilled water. Run in from a burette standard N/10 sodium thiosulphate solution until the solution is straw yellow, add soluble starch indicator or methylene blue and complete the titration.

The use of acetic acid instead of hydrochloric acid as generally given in manuals is recommended for the elimination of an error due to the possible presence of chlorates in the bleach. In the event of chlorates being present titration with thiosulphate in the presence of hydrochloric acid will give results which are too high.

Method B.

Introduce the 20 cc. portion of the sample into a 250 cc. Erlenmeyer flask. Add about 80 cc. distilled water, mix well and run in from a burette standard N/10 sodium arsenite solution until a drop removed and placed on potassium iodide starch paper gives no blue color.

Or the standard arsenite may be added in excess, soluble starch indicator added and standard N/10 iodine solution run in from a burette until a blue color appears or if methylene blue is used until the blue color disappears.

¹ Sutton, Vol. Anal. 9th Edition, p. 173.

APPENDIX A.

In the Laboratory Course in Sanitary Chemistry given at Cornell University, seven and one-half hours per week for a period of fifteen weeks are devoted to Water Analyses. The authors have found that the student can utilize his time to best advantage by having each week two laboratory periods of three and three-quarters hours each. In accordance with this practice the following schedule of laboratory work to be completed each week has been formulated.

First Week. Prepare the following solutions:—

“Cleaning Mixture,” page 29.

Standard Hydrochloric Acid, N/10, page 9.

Standard Sulphuric Acid, N/10 and N/50, page 11.

Standard Sodium Hydroxide, N/10, page 11.

Standard Potassium Permanganate, Solution A,
page 15.

Standard Sodium Chloride, page 13.

Water for Standard Silver Nitrate, page 13.

Standardize and adjust N/10 Hydrochloric Acid, page 9.

Second Week. Prepare the following solutions:—

Standard Sodium Carbonate, N/22, page 16.

Standard “Soda Reagent,” N/10, page 12.

Standard Silver Nitrate, page 13.

Standard Oxalic Acid, page 16.

Standard Iron, page 21.

Standard Potassium Permanganate, Solution B,
page 16.

Standardize and adjust:—

N/10 Sodium Hydroxide, page 11.

N/10 and N/50 Sulphuric Acid, page 11.

Third Week. Prepare the following solutions:—

Potassium Chromate, page 27.

Nessler Reagent, page 22.

Potassium Hydroxide for Oxygen Dissolved, page 24.

Sulphuric Acid for Oxygen Dissolved, page 24.

Sulphuric Acid 1:4, page 24.

Alkaline Permanganate, page 24.

Standard Sodium Thiosulphate, page 19.

Standard Primary Potassium Sulphate, page 21.

Standard Potassium Dichromate (A) for SO_4 , page 19; and (B) for standardizing thiosulphate solutions, page 19.

Standardize and adjust:—

N/22 Sodium Carbonate, page 16.

N/10 "Soda Reagent," page 12.

Potassium Permanganate, A and B, page 15.

Fourth Week. Prepare the following solutions:—

Saturated Sodium Carbonate, page 25.

Nitrite Reagent, page 23.

Potassium Hydroxide for Nitrates, page 26.

Nitric Acids for Mn (two solutions), page 25.

Sulphuric Acid for Mn, page 25.

Standard Potassium Nitrate, page 18.

Standard Potassium Nitrite, page 17.

Standard Sodium Phosphate, page 21.

Standardize and adjust:

Silver Nitrate, page 13.

Oxalic Acid, page 16.

Sodium Thiosulphate, page 19.

Primary Potassium Sulphate, page 21.

Fifth Week.

Prepare and standardize the following solutions:

Standard Iodine, N/10, page 18.

Standard Arsenite, N/10, page 20.

At the end of the fifth week all of the apparatus which will be used in the chemical examination of water must have been thoroughly cleaned with cleaning mixture, rinsed and dried, the distillation apparatus for the determination of ammonia must have been set up, and all the solutions required in the work must have been made and standardized.

Sixth and Seventh Weeks.

A complete chemical examination of a polluted surface water.

Because, at the start, the student is unfamiliar with the methods and will in consequence work less rapidly than later in the course, two weeks are devoted to this analysis, and the following division of the work is suggested. As many determinations as possible should be kept going at the same time.

Sixth Week.

Total Solids
Free CO₂
Appearance
Color
Odor
Turbidity
Alkalinity
Total Hardness
Mineral Acid Hardness
N as Free Ammonia
N as Total Ammonia
N as Nitrites
N as Nitrates
Chlorine
Loss on Ignition

Seventh Week, first period.

Oxygen dissolved
Oxygen consumed

Iron (Fe)

Manganese (Mn)

Phosphates (PO_4)

Seventh Week, second period.

Solids in solution

Solids in suspension

Coefficient of fineness

Sulphates (SO_4)

Cleaning and plugging of tubes for bacteriological media.

Eighth Week.

Preparation, tubing and sterilizing of culture media for bacteriological work. The amounts of media to be tubed will be posted on the bulletin board.

Ninth Week.

Bacteriological Examination of Water. The number of tubes of each kind of media to inoculate will be posted.

Tenth Week.

Chemical and Bacteriological Examination of a Water Sample.

In the chemical analysis, the order of procedure recommended on p. 70 is advised. In the bacteriological analysis, all the inoculations must, of necessity, be made during the first period, a list of the inoculations to be made will be posted on the bulletin board.

Eleventh and Twelfth Weeks.

Chemical and Bacteriological examinations of various water samples.

Studies of presumptive tests for *B. coli*.

Filter control methods.

Thirteenth, Fourteenth and Fifteenth Weeks.

Boiler water analysis.

Special phases of sanitary water examination.

APPENDIX B.

INTERNATIONAL ATOMIC WEIGHTS, 1910.
 Abridged Table from J. Am. Chem. Soc. 32, 1.

	Symbol	Atomic Weight		Symbol	Atomic Weight
Aluminum	Al	27.1	Lithium	Li	7.0
Antimony	Sb	120.2	Magnesium	Mg	24.32
Arsenic	As	74.96	Manganese	Mn	54.93
Barium	Ba	137.37	Mercury	Hg	200.0
Bismuth	Bi	208.0	Molybdenum	Mo	96.0
Boron	B	11.0	Nickel	Ni	58.68
Bromine	Br	79.92	Nitrogen	N	14.01
Calcium	Ca	40.09	Oxygen	O	16.0
Carbon	C	12.0	Phosphorus	P	31.0
Chlorine	Cl	35.46	Platinum	Pt	195.0
Chromium	Cr	52.	Potassium	K	39.1
Cobalt	Co	58.97	Silicon	Si	28.3
Copper	Cu	63.57	Silver	Ag	107.88
Fluorine	F	19.0	Sodium	Na	23.
Gold	Au	197.2	Strontium	Sr	87.62
Hydrogen	H	1.008	Sulphur	S	32.07
Iodine	I	126.92	Tin	Sn	119.0
Iron	Fe	55.85	Zinc	Zn	65.37
Lead	Pb	207.10			

TABLE OF REACTING WEIGHTS OF RADICLES FOR INDUSTRIAL
 WATER COMPUTATIONS.

Computed from Atomic Weights of 1910.

	Symbol	Reacting Weight	Logarithm*
Aluminum	Al	0.1107	.04415
Bicarbonate	HCO ₃	0.0164	.21484
Calcium	Ca	0.0499	.69810

*Symbol Reacting Weight Logarithm**

Carbonate	CO ₃	0.0333	.52244
Chloride	Cl	0.0282	.45025
Hydrogen	H	0.9920	.99651
Iron	Fe''	0.0358	.55388
Magnesium	Mg	0.0822	.91487
Nitrate	NO ₃	0.0161	.20683
Phosphate	PO ₄	0.0316	.49969
Potassium	K	0.0255	.40654
Sodium	Na	0.0434	.63749
Sulphate	SO ₄	0.0208	.31806

Factors

<i>Found</i>	<i>Wanted</i>	<i>Factor</i>	<i>Logarithm *</i>
Acidity in HCl	H	0.0276	.44091
H ₂ SO ₄	H	0.0206	.31387

TABLE GIVING GRAMS HCl IN 100 cc. OF HCl OF DIFFERENT
DENSITIES AT 15°C.

Lunge and Marchlewski.

Specific Gravity	Grams HCl in 100 cc.	Specific Gravity	Grams HCl in 100 cc.
1.095	20.9	1.150	34.0
1.100	22.0	1.155	35.3
1.105	23.2	1.160	36.6
1.110	24.3	1.165	37.9
1.115	25.5	1.170	39.2
1.120	26.7	1.175	40.4
1.125	27.8	1.180	41.8
1.130	29.1	1.185	43.0
1.135	30.3	1.190	44.3
1.140	31.5	1.195	45.6
1.145	32.8	1.200	46.9

* Characteristics are omitted.

TABLE GIVING GRAMS SO_2 IN 100 cc. OF H_2SO_4 OF DIFFERENT DENSITIES AT 15°C .

Lunge and Isler.

Specific Gravity	Grams SO_2 in 100 cc.	Specific Gravity	Grams SO_2 in 100 cc.
1.65	98.1	1.75	116.5
1.66	99.8	1.76	118.5
1.67	101.6	1.77	120.4
1.68	103.4	1.78	122.8
1.69	105.3	1.79	125.2
1.70	107.1	1.80	127.7
1.71	108.9	1.81	130.5
1.72	110.8	1.82	133.8
1.73	112.7	1.83	137.6
1.74	114.6	1.84	143.6

TABLE GIVING GRAMS HNO_3 IN 100 cc. OF HNO_3 OF DIFFERENT DENSITIES AT 15°C .

Lunge and Rey.

Specific Gravity	Grams HNO_3 in 100 cc.	Specific Gravity	Grams HNO_3 in 100 cc.
1.20	38.8	1.33	69.7
1.21	40.9	1.34	72.5
1.22	43.0	1.35	75.3
1.23	45.2	1.36	78.3
1.24	47.5	1.37	81.4
1.25	49.8	1.38	84.6
1.26	52.1	1.39	87.9
1.27	54.4	1.40	91.4
1.28	56.8	1.41	95.2
1.29	59.3	1.42	99.1
1.30	61.7	1.43	103.2
1.31	64.3	1.44	107.5
1.32	66.9	1.45	112.1

TABLE GIVING GRAMS NH_3 IN 100 cc. OF NH_4OH OF
DIFFERENT DENSITIES AT 15°C .

Lunge and Wernik.

Specific Gravity	Grams NH_3 in 100 cc.	Specific Gravity	Grams NH_3 in 100 cc.
.936	15.74	.916	21.09
.934	16.27	.914	21.63
.932	16.81	.912	22.19
.930	17.34	.910	22.74
.928	17.86	.908	23.29
.926	18.42	.906	23.83
.924	18.93	.904	24.39
.922	19.47	.902	24.94
.920	20.01	.900	25.50
.918	20.56	.898	26.05

APPENDIX C.

USEFUL TABLES, FACTORS AND CONVERSION UNITS FOR WATER COMPUTATIONS.

MEASURES OF LENGTH.

- 1 inch = 0.08333 ft. = 2.54 cm.
 1 foot = 30.48 cm. = 0.30479 meter.
 1 yard = 0.9144 meter.
 1 mile = 5,280. ft.
 1 mile = 1,609.31 meters.
 1 rod = 16.5 ft. = 5.028 meters.
 1 chain = 66. ft. = 20.1161 meters.
 1 fathom = 6. ft. = 1.829 meters.

- 1 millimeter = 0.03937 inch.
- 1 centimeter = 0.3937 inch.
- 1 meter = 39.3709 inches.
- 1 kilometer = 0.6214 mile.

MEASURES OF AREA.

- 1 square inch = 645.137 sq. mm.
- 1 square foot = 928.997 sq. cm.
- 1 square yard = 0.836 square meter.
- 1 acre = 160 sq. rods = 4,840. sq. yards = 43,560 sq. ft.
- 1 acre = 4,046.87 sq. meters.
- 1 square mile = 640. acres = 2. 5899 sq. kilometer.

-
- 1 square millimeter = 0.00155 sq. inch.
 - 1 square centimeter = 0.15501 sq. inch.
 - 1 square meter = 10.7643 sq. ft. = 1.1960 sq. yds. = 0.00025 A.
 - 1 square kilometer = 0.386 sq. mile.

MEASURES OF VOLUME AND CAPACITY.

- 1 cubic inch = 16.387 cubic cent.
 - 1 cubic foot = 28.32 liters.
 - 1 cubic yard = 0.765 cubic meter.
-
- 1 liter = 61.023 cu. in.
 - 1 liter = 0.0353 cu. ft.
 - 1 cc. = 0.061 cu. in.
-
- 1 fluid ounce (U. S.) = 456 grains = 1.8044 cu. in.
 - 1 fluid ounce (U. S.) = 29.57 cc.
 - 1 pint = 16. fl. oz. = 473.125 cc.
 - 1 quart = 57.75 cu. in. = 946.36 cc.
 - 1 gallon (U. S.) = 231. cu. in. = 3.785 L.
 - 1 gallon (Eng.) = 277. cu. in. = 4.5435 L.
 - 1 barrel (wine meas.) = 31.5 gallons = 119.228 L.
 - 1 barrel (beer meas.) = 36. gallons = 137.26 L.
 - 1 bushel = 2,150.42 cu. in. = 35.24 L.

MEASURES OF WEIGHT.

- 1 ounce Troy = 480 grains = 31.104 grams.
 1 ounce Avd. = 437.5 grains = 28.347 grams.
 1 pound Troy = 12 oz. = 5,760 grains = 373.242 grams.
 1 pound Avd. = 16 oz. = 7,000 grains = 453.592 grams.
 1 Ton (2000 lb.) = 907.2 kilos.
 1 gram = 15.432 grains.
 1 kilo = 2.6786 lb. Troy.
 1 kilo = 2.205 lb. Avd.

WEIGHTS AND VOLUMES OF WATER.

- 1 gallon (U. S.) = 58,380. grains = 8.345 lbs. Avd. = 3.785 kilos.
 1 gallon (Eng.) = 70,000 grains = 10. lbs. Avd. = 4.543 kilos.
 1 cubic foot = 7.48 U. S. Gals. = 28.315 kilos.
 1 cubic foot = 62.425 lbs. Avd.
 1 cubic inch = 252.88 grains = 0.0367 lb. Avd. = 16.387 gms.
 1 liter = 0.2642 U. S. Gals. = 2.205 lb. Avd. = 61.027 cu. in.
 1 pound Avd. = 27.6814 cu. in. = 0.016 cu. ft. = 0.1198 U. S. Gals.
 1 pound Avd. = 453.593 cc. = 0.454 L.

APPROXIMATE RATES OF FLOW, ETC., OF WATER.

- 1 inch vertical velocity per hour = 0.62 U. S. Gal. per sq. ft. per hr.
 1 foot vertical velocity per hour = 7.44 U. S. Gal. per sq. ft. per hr.
 1 U. S. Gal. per sq. ft. per hr. = 1.60 in. vertical velocity per hour or 4.07 cm.
 1 cu. ft. per second = 448.8 gals. per min.
 1 cu. ft. per second = 26,928 gals. per hour.
 1 cu. ft. per second = 646,272. gals. per day.

- 1 cu. ft. per second = 1 in. per A. per hr.
- 1 U. S. Gal. per second = 0.01338 cu. ft. per sec.
- 1 cubic meter per min. = 0.5886 cu. ft. per sec.
- 1 U. S. Gal. per sq. ft. per hr. = 1.045 million gals. per Acre per day.
- 1 U. S. Gal. per sq. ft. per hr. = 1.2 cu. ft. per sq. yd. per hr.
- 100,000 gals. per day = 69.4 gals. per min.
- 1 million gals. per day = 41,667. gals. per hr. = 694. gals. per min.

APPROXIMATE PRESSURES AND WORK.

- 1 pound per sq. in. = 2.31 ft. water column.
- 1 foot elevation water column = 0.5 lb. per sq. in. pres. (allowing for friction).
- 1 horse power = 1 cu. ft. per second falling 8.8 ft.
- 1 horse power = 550 foot pounds per sec.
- 1 H.P. of Boiler = Evap. 30 lbs. per hr. at 70 lbs. per sq. in. from 100°F.
- 1 cu. ft. evap. at atmospheric pres. = 1 cu. ft. steam.
- 1 Atmosphere = 760 mm. Hg = 29.9 in. Hg = 33.9 ft. water = 14.7 lbs. per sq. in. = 1033.3 grams per sq. cm.
- 1 Boiler Horse Power requires approximately 30 lbs. (3.59 gals.) water evaporated per hour.
- 1 Indicated H.P. requires in large condensing engines about 1.75 U. S. Gallons water evaporated per hour.
- 1 Indicated H.P. requires in small non-condensing engines, often 7 to 8 U. S. gallons water evaporated per hour.
- 1 Pound Coal will evaporate approximately 1 gallon of water.
- 1 Pound of Steam requires 20 lbs. (2.39 gals.) water 60°F. to condense in surface condensers.

MISCELLANEOUS FACTORS, UNITS, AND FORMULAS.

Grains per U. S. Gallon = Parts per million \times 0.0584.

Parts per Million = Grains per U. S. Gallon \times 17.1.

1 minute = 0.01666 hr.

Friction in pipes proportional to square diameter.

Capacity pipes increases as square diameter.

Capacity Pump Cylinder = $\frac{\text{Area} \times \text{stroke}}{231}$ = gallons.

To obtain 1 grain per gal. per mil. gals. = requires 72 gals.
per hr. of 1% sol.

A 1% solution = 583.8 grains per U. S. Gallon.

Diameter Circle = circumf. \times 0.31831.

Circum. Circle = Diam. \times 3.1416.

Area Circle = Diam.² \times 0.7854.

Surface Sphere = $4 \pi r^2$.

Surface Segment = $2 \pi rh$.

Volume Sphere = $D^3 \times 0.5236$.

Area Sector $N^\circ = \frac{N}{360} r^2 \pi$.

Area Segment $N^\circ = \frac{r^2}{2} \left(\frac{N}{180} \pi - \sin N \right)$.

Area ellipse = $\pi a b$ (a, b, semiaxes).

Area Parabola Segment = $\frac{2}{3} CL$ (c = chord, L = height
of arc).

Volume Cone or Pyramid = $\frac{1}{3}$ base \times alt.

Volume Frustum = $\frac{h}{3} (B + \sqrt{Bb} + b)$. B = area base,
b = area top, h = dist. B to b).

THERMOMETRIC AND SPECIFIC GRAVITY EQUIVALENTS.

$$F^{\circ} \text{ to } C^{\circ} = (F^{\circ} - 32) \times 0.555.$$

$$C^{\circ} \text{ to } F^{\circ} = 1.8^{\circ}C + 32.$$

$$F^{\circ} \text{ to } R^{\circ} = (F^{\circ} - 32) \times 0.444.$$

$$R^{\circ} \text{ to } F^{\circ} = 2.25 F^{\circ} + 32.$$

$$R^{\circ} \text{ to } C^{\circ} = 1.25 C^{\circ}.$$

$$C^{\circ} \text{ to } R^{\circ} = 0.8 R^{\circ}.$$

$$\text{Sp. Gr.} = \frac{140}{B^{\circ} + 130} \text{ for Baumé lighter than water.}$$

$$B^{\circ} = \frac{140}{\text{Sp. Gr.}} - 130 \text{ for Baumé lighter than water.}$$

$$\text{Sp. Gr.} = \frac{145}{145 - B^{\circ}} \text{ for Baumé heavier than water.}$$

$$B^{\circ} = 145 \frac{145}{\text{Sp. Gr.}} \text{ for Baumé heavier than water.}$$

APPENDIX D.

The following questions may serve as a Syllabus of Chemistry, Course 75, Potable and Industrial Water (Lectures, 2 hours a week).

The examination in this course will be based upon questions selected from this list and from such others as may be posted upon the bulletin board.

Problems involving the application of water analyses to municipal and industrial waters and to water purification will be issued on mimeographed sheets.

1. Explain what is meant by the terms: potable water, wholesome water, infected water, polluted water.
2. What are the six most important kinds of water examinations an analyst is called upon to make with reference to the different uses to which the water is destined?
3. What constitutes a satisfactory water supply in the light of our present knowledge?
4. What sort of information or data will be comprised in the record of the "History of the Sample" or "environmental data"? Explain why it is impossible for an analyst to intelligently interpret the results of an analysis without such information.
5. To what class of objectionable materials in water is the term "impregnated" water applied?
6. What are the sources of atmospheric water supplies?
7. How may these supplies become polluted? Infected?
8. State some of the characteristics of rain water.
9. What information should an analyst obtain prior to making an analysis of rain water collected and stored in a cistern? Well water? Spring water?
10. State some of the causes for the failure of an artesian supply. Cite cases in point.
11. Name the chief agencies leading to the natural or self-purification of surface waters.
12. Discuss these agencies and the results accomplished.
13. Why is there reason to believe that the amount of purification ascribed to running water is over estimated and is more apparent than real?
14. Of what importance are water temperature measurements?
15. How are these measurements made? Describe the principles of the instruments used.

16. Define: "Direct stratification," "Inverse stratification," "thermocline," "spring circulation," "great overturning," "periods of stagnation."

17. Describe the cycle of seasonal movements of the waters of a lake or pond due to temperature changes.

18. What are the chief sources of ground water supplies?

19. What is an artesian well?

20. Name the most important agencies leading to the purification of a water as it sinks through the soil.

21. Distinguish between velocity and flow in ground waters.

22. What is meant by "effective size" and "uniformity coefficient" of a sand? Why was the term "effective size" suggested by Hazen?

23. What is meant by pressure gradient?

24. Define "transmission constant."

25. Give approximate velocity and flow for "fine sand."

26. Name the chief agencies or causes of pollution of ground water.

27. How do most of the shallow wells giving rise to typhoid fever become infected?

28. What is the Water Table? What relation does it bear to surface contour?

29. Make a sketch showing proper arrangement of pump and well drawing from a ground water level of but little depth.

30. Upon what four factors does the quantity of ground water that may be pumped from driven wells, chiefly depend?

31. Describe the American standard for color.

32. Name three methods for measuring color.

33. Of what importance are color measurements?

34. Cite instances where color measurements are constantly used by analysts as an aid in other work.

35. What is meant by turbidity? By coefficient of fineness?

36. What is the standard unit of turbidity?

37. Give three methods for the determination of turbidity.

38. What is meant by "Free Ammonia"? How is it determined?

39. What by "Albuminoid Ammonia"? What by "Total Organic Nitrogen"? How are these determined?

40. Why is it as important to record in the note book the rate of the distillation of the ammonia as the total amount found?

41. Discuss the significance of "Free Ammonia" and "Albuminoid Ammonia" in surface waters. In ground waters. In atmospheric waters.

42. What seasonal fluctuations in ammonias are generally observed in stored waters? Why?

43. Is it possible to set "local standards" for ammonias? Discuss.

44. Approximately how much free ammonia and how much albuminoid ammonia will average American fresh sewage contain?

45. How much of these two forms of nitrogen does each inhabitant contribute to the municipal sewage in twenty-four hours? What practical use is made of these figures?

46. State the composition of Nessler's reagent and give the formula generally assigned to the brown compound formed when the reagent is added to ammoniacal solutions.

47. How may the quantity of nitrogen as nitrite in a water be determined? Nitrogen as nitrate?

48. Write reactions involved in the determination of nitrites; of nitrates?

49. What are the sources of nitrites in water? What is the interpretation of the results obtained?

50. Discuss the source and significance of nitrates in surface waters; in ground waters.

51. From what unusual sources may nitrites find their way into water supplies?

52. State some of the important sources of error in the determination of nitrites; of nitrates.

53. How is chlorine determined?

54. Of what value is the determination of chlorine? Illustrate by example.

55. What are the chief sources of error in the method and how may they be avoided?

56. How much chlorine does average American sewage contain?

57. Describe the methods of ascertaining the approximate evaporation of a boiler by means of chlorine determinations.

58. Describe a method for the approximate measurements of the velocity of underground water by means of chlorine determinations.

59. What is meant by "oxygen consumed"? How is it determined?

60. What is the significance of high oxygen consumed in a ground water? In a surface water?

61. What are the reducing substances which may be present in water and giving rise to "oxygen consumed"?

62. How are total solids determined?

63. How solids in suspension (give two methods)?

64. What is meant by "loss on ignition"? Of what value is the determination? To what is the loss due?

65. What is meant by "dissolved oxygen"? Give briefly two methods by which it may be measured.

66. Of what use are determinations of "dissolved oxygen"?

67. Give methods for taking samples of water for this determination.

68. Cite instances where the determinations of dissolved oxygen have been of material assistance in the solution of water supply problems.

69. What relation exists between the amount of oxygen in solution and the depth at which the sample is taken? How will the values differ according to the season of the year?

70. What is Levy's "coefficient of alterability" and what did he believe it indicated?

71. What is meant by the "relative stability of an effluent"? How is this value determined? What is its significance?

72. In what form does carbon dioxide exist in water?

73. Describe briefly the methods in use for the determination of carbon dioxide in its different combinations.

74. When a natural water is alkaline to phenolphthalein what does it probably contain?

75. How would you ascertain the presence and the amount of normal sodium carbonate in water? Of calcium hydroxide?

76. What is meant by the hardness of a water? What by alkalinity? What by mineral acid hardness? In what units are these terms expressed?

7. Upon what does the soap consuming power of a water depend? Write an equation for a reaction showing soap consumption. ✓

78. Explain Whipple's formula for the depreciation

H

in value of a water supply due to hardness, $D = \frac{H}{10}$.

79. State under what conditions we usually find the hardest waters. Explain why.

80. Why is the Clark soap method unreliable as a means of measuring hardness?

81. Of what value is a determination of the amount of phosphates in a water?

82. What are the sources of phosphates?

83. How are phosphates determined in a sanitary analysis? What is the chief source of error in the method?

84. When are determinations of the sulphate content of a water necessary? How may sulphates be determined? Write equation for all reactions in the colorimetric determination.

85. How do we determine the quantity of iron in a water? In what form does iron generally exist in water?

86. What are the objections to the presence of iron in water? What class of waters are often high in iron?

87. What organism gives rise to trouble in iron containing waters? Why?

88. How is iron generally removed from water intended for municipal use?

89. What is meant by "Local Standards"? What are their uses? Their limitations?

90. What are "iso-chlors"? How are they plotted? What is their value?

✓ 91. Name the diseases of man which may be water-borne. Of animals.

92. Cite cases of epidemics other than typhoid which are believed to have been water-borne.

✓ 93. What is meant by "normal typhoid death-rate"? By what other terms is this also known? How is this value estimated? What is the "normal" generally assigned to municipalities using ground waters? Surface waters? Filtered waters?

94. Name and describe briefly three epidemics of typhoid fever the result of an infected ground water sup-

ply. Three from infected surface water. Three from failure in management or controls of purification plants.

95. Explain Whipple's formula for depreciation due to sanitary quality $D = 2.75 (T-N)$.

96. What is meant by "Typhoid carriers"?

97. What is meant by "secondary infection"? How is the number of such cases in an epidemic estimated?

98. How long is it believed that *B. typhosus* will live in pure water? In polluted water? In sewage?

99. Cite instances tending to prove that *B. typhosus* may be carried long distances by water. May live for long periods in soil.

100. In order that a typhoid epidemic may be considered as proved to be due to the water supply, what data are essential?

101. Why do water analysts lay so much emphasis on the detection of the presence in water of organisms of the *B. coli* group? ✓

102. What other groups of bacterial organisms are indications of pollution in water?

103. What is the significance of a preponderance of micrococci in a water sample? Of a relatively large number of moulds?

104. Name several species of "filamentous bacteria" found in waters with their characteristics and significance.

105. State briefly the reasons why it is generally impracticable to attempt to isolate *B. typhosus* from a water sample and why this is not essential for condemning the water as unfit for household use. ✓

106. State briefly the successive steps in the cycle of changes through which the nitrogenous material in water probably passes, and show how the final nitrogen compounds found in a water may be used to throw light upon the present, past and probable future character of the water.

107. What is meant by the "plankton" of a water supply?

108. Describe the Sedgwick-Rafter-Whipple method for the quantitative measurement of the microscopic organisms in water.

109. In a supply drawn from small lakes or large storage reservoirs at what seasons of the year do we expect to find the largest numbers of (a) Diatomaceae, (b) "Green Algae," (c) Protozoa, (d) "Blue-green Algae"? Why?

110. What are the most important causes of bad odors and tastes in surface waters? In ground waters?

111. Name five organisms giving rise to disagreeable odors and tastes.

112. What class of organisms are usually responsible for fishy odors, "pig pen," "grassy"?

113. What are the adjectives used to record odors indicated by the following abbreviations—v., a., e., f., m., M., d., p., s.?

114. What are the odor intensities indicated by the numerals 1, 2, 3, 4, 5, when placed before these letters?

115. How are "hypothetical combinations" calculated in boiler water analyses?

116. Why is it preferable to report results in ionic form rather than combined as hypothetical salts?

117. Of what use is bark extract or tannin in a scale preventive?

118. When is lime used in softening water? When soda ash?

119. Why is a water depositing a very little "scale" a better water for steam generation than one which deposits none?

120. What is the objection to "hard waters" for use in steam boilers?

121. What is "scale"? How is it formed? How may it be prevented?

122. What is the difference in general composition between a soft and a hard scale?

123. What is meant by "priming"; by foaming? To what sort of troubles do these phenomena give rise? What is the cure?

124. How may we ascertain by analysis whether a water is probably a "foaming" water?

125. What substances present in a water cause corrosion? Explain.

126. What constituents enter into most of the commercial scale preventatives?

127. How may water be softened? How far is it practicable to carry the softening process? ✓

128. Describe the component parts of a modern slow sand water purification plant, explaining the function of each part.

129. State the opinion held at the present time, explaining the mechanism of the action of the slow sand filter in removing bacteria.

130. At what rates per A. per day are slow sand filters commonly operated?

131. What is meant by "scaping" a filter? How is this accomplished? How often must this be done? How much material is removed? What is the customary procedure as to the number of times a filter bed may be thus treated without replacement of sand?

132. Describe the operation of placing a slow sand filter in service.

133. How does the operator ascertain when a filter effluent is of proper character to deliver into the service mains?

134. Give maximum and minimum depths of sand usually adopted in slow sand filter beds. Of gravel.

135. Why are filter beds covered?

136. What are "preliminary filters" or "scrubbers"? Describe some of the types of construction in use. What do they accomplish?

137. What is meant by the "effective size" of filter sand? How is it determined? Of what use is this value? Cite examples of effective size in use in American or European plants.

138. What sort or nature of sand is supposed to give the best results in slow sand beds?

139. Why in filling a sand bed is water usually introduced from below?

140. What is meant by "loss-of-head"? How is the loss-of-head used in the control of a filter plant?

141. Describe one or more types of sand removing machines, sand washing machines, re-sanding machines.

142. What are under drains? How are they constructed? Why is considerable engineering skill required for the proper construction and laying out of an under-drain system?

143. Explain why a filter bed will seldom deliver a satisfactory effluent continuously, if the head of water on the filter is variable.

144. State briefly the tests or determinations which should be made by the analyst in control of a water purification plant, giving reasons why such tests or determinations are made.

145. Upon what factors or conditions in general does the efficiency of a slow sand filter depend?

146. What types of purification plants are meant by "American," "rapid" or "mechanical" filtration systems?

147. What are "coagulants"? How are they applied? What are their functions? What do they accomplish? Name some of those in use.

148. What should be the approximate composition of a good aluminum coagulant?

149. State the component parts of a typical rapid water filtration plant.

150. What are "strainers"? Name and describe several varieties. How are they placed? What is their function?

151. What are coagulation basins? What are their functions? Of what value are they? How are they cleaned? Describe two types.

152. The use of coagulants requires the presence of what components or constituents in a water? Suppose such constituents are absent, what must be done?

153. How may the operator decide upon the quantity of coagulant to apply? How ascertain whether any undecomposed coagulant is passing into the filter effluent?

154. What is meant by "red-water"? What explanations are given for its cause? May it be remedied?

155. What are the so-called "lime-bacteria"? Of what importance are they?

156. Describe the method of cleaning two different types of rapid system filters.

157. How does the operator ascertain when a filter should be washed?

158. What is the advantage of stirring the sand mechanically during washing? What systems are in use for the purpose?

159. What may be considered as a fairly average cost of filtration by the American or rapid system? By the slow sand method?

160. The filtration with the use of coagulants is essential for the proper purification of what sorts of waters?

161. Describe in detail the water purification plant of the City of Ithaca.

162. What efficiency may be expected of a rapid water filtration plant?

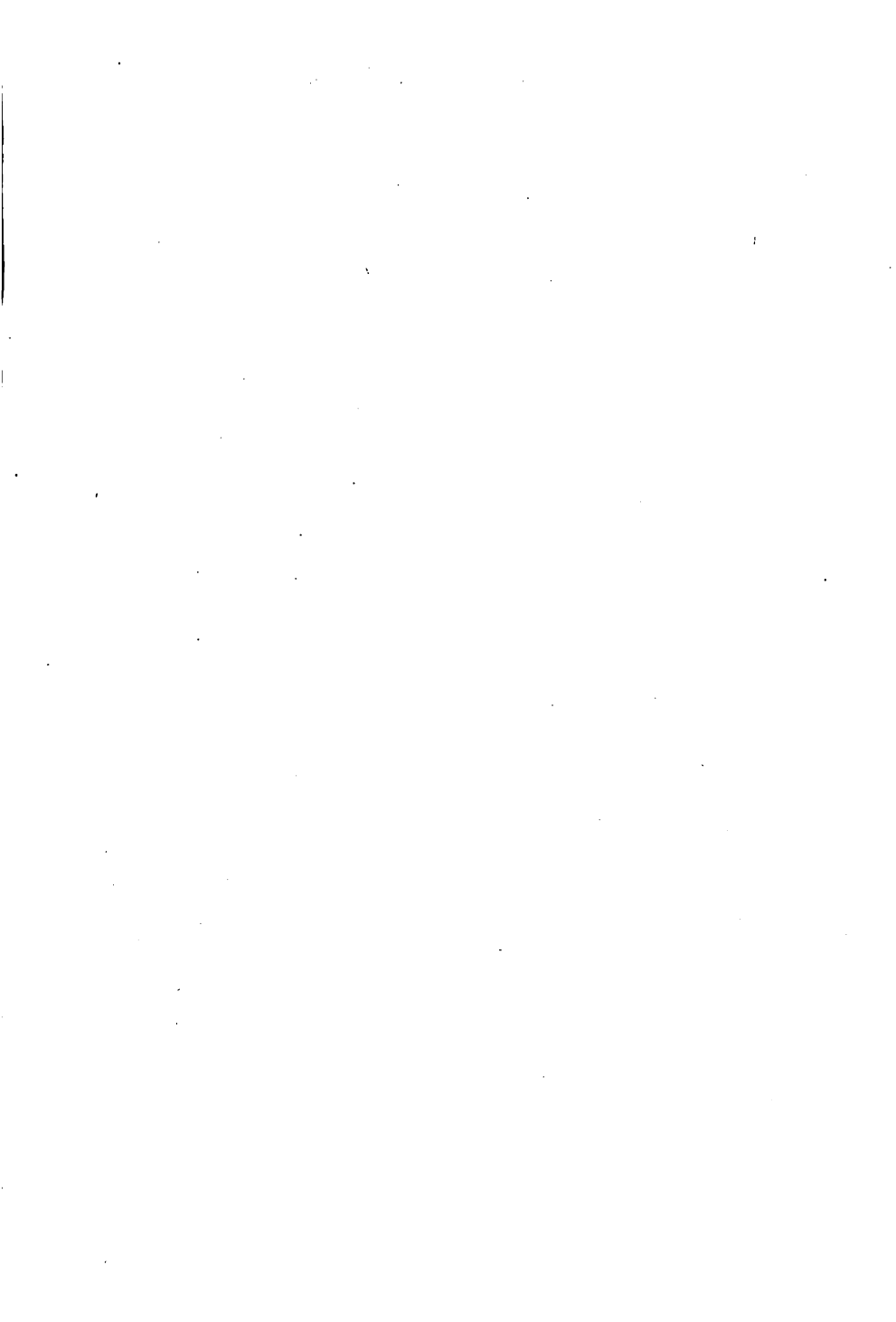
163. Describe the present types of systems of water purification by Ozone. What are the chief difficulties met with? How efficient are these processes? Compare their efficiencies and cost of treatment with the filtration systems.

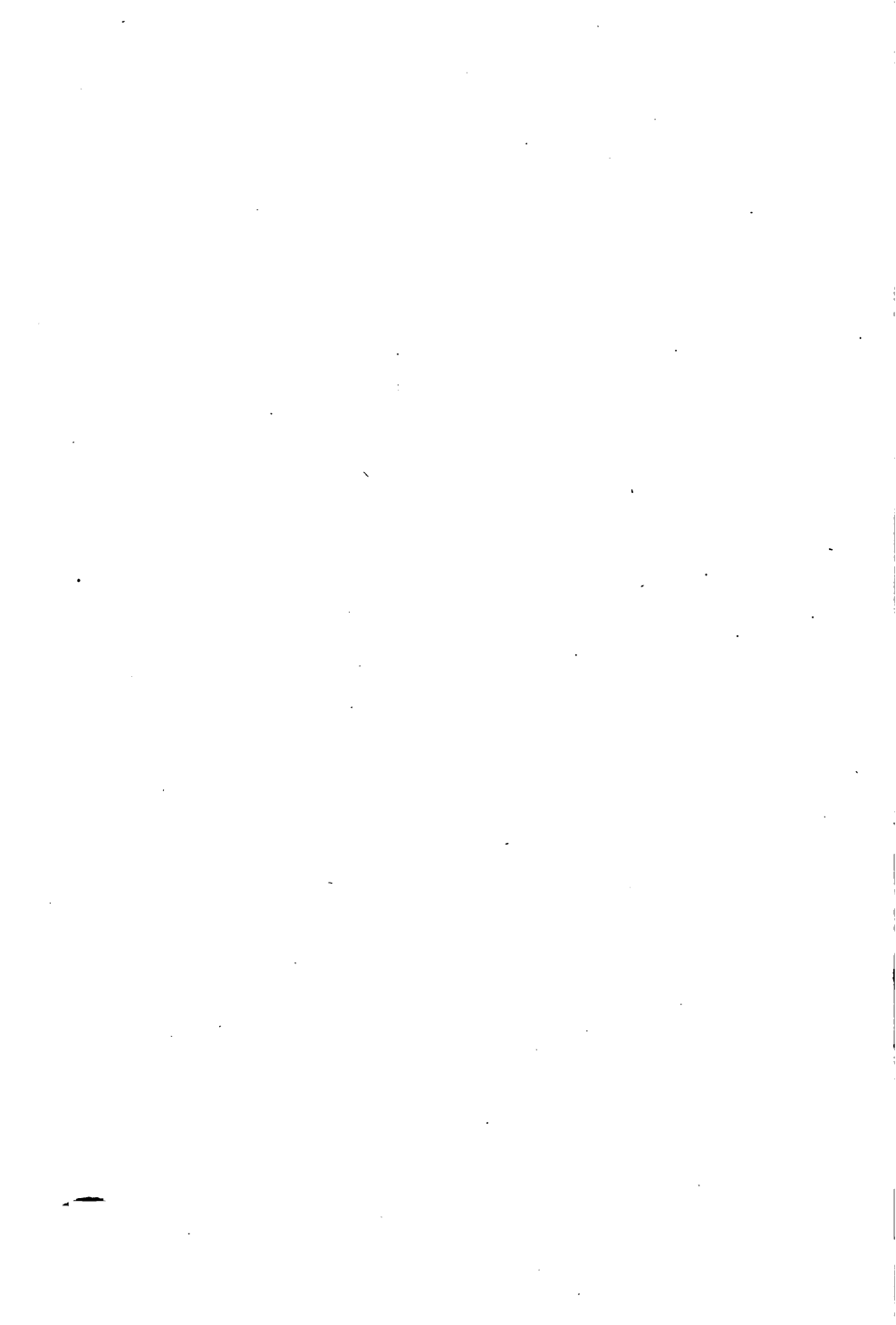
164. How is ultra-violet light being applied in water purification? What sort of installation is required? What kind of waters may be treated?

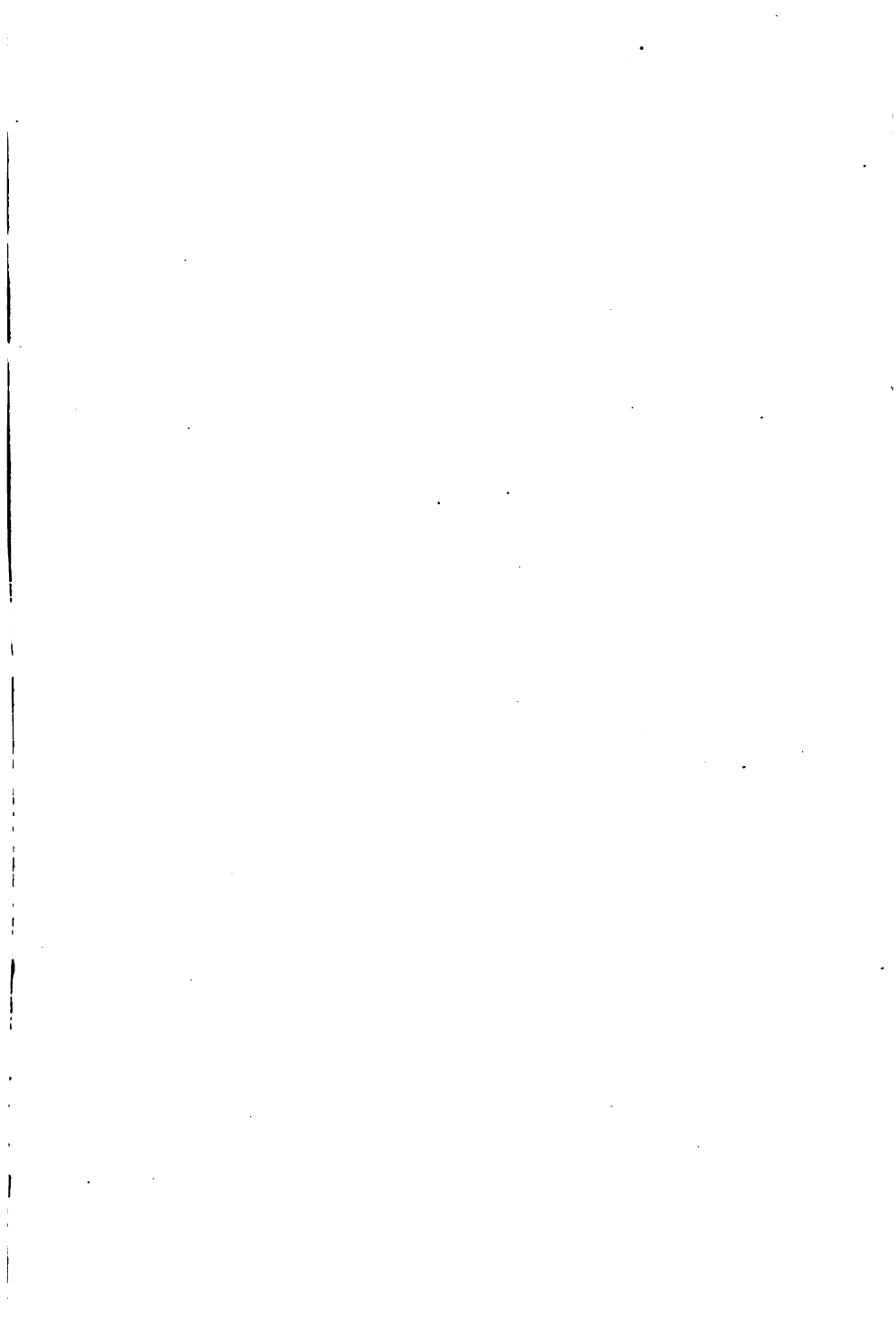
165. Describe the use of hypochlorites in the purification of waters. What are some of the good and bad points of this method? What kinds of waters are usually unsuccessfully treated? What is meant by available chlorine? Explain the reactions taking place when the hypochlorite acts in water. Why has the term "potential oxygen" been suggested as being more specific than that of available chlorine?

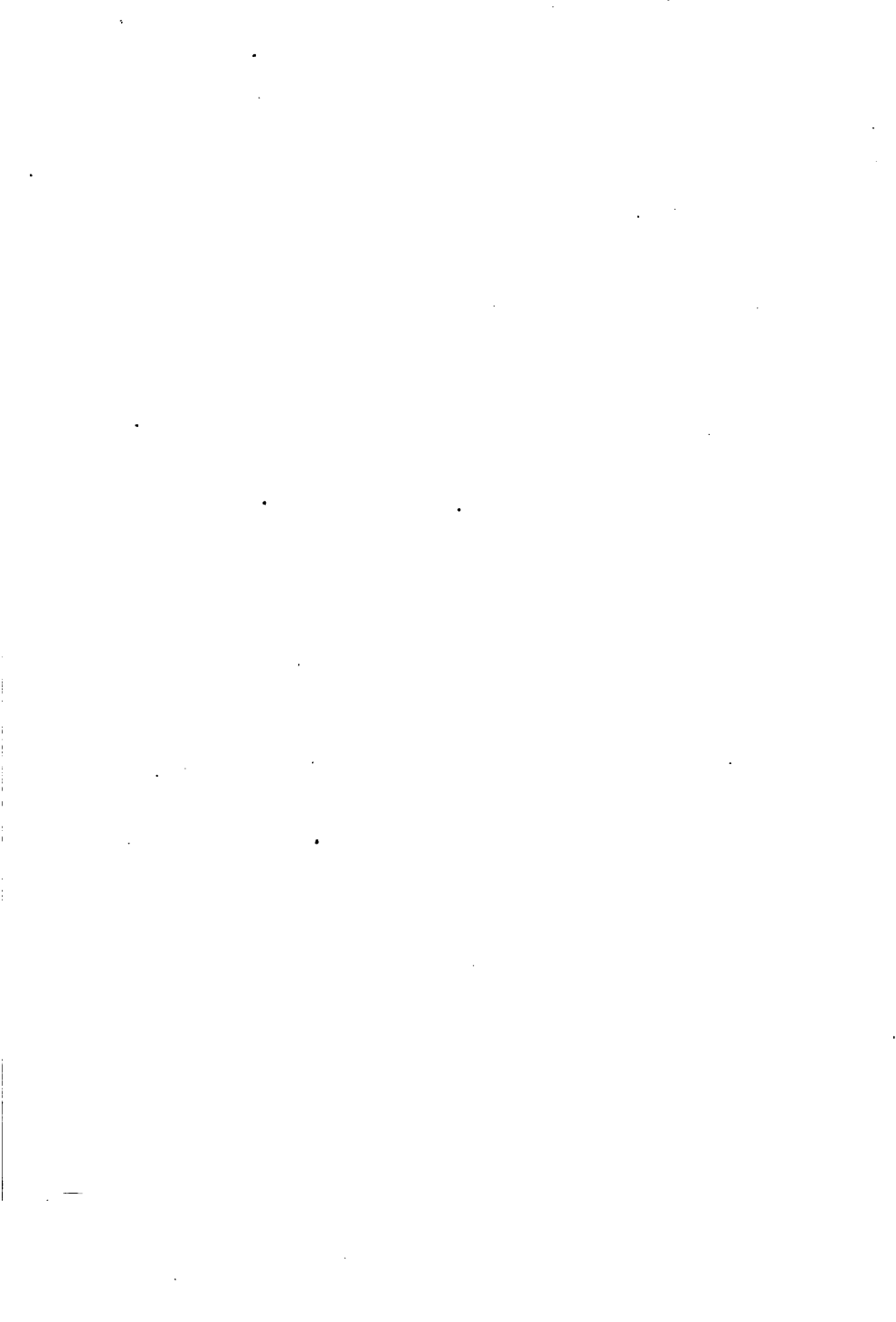
166. Describe a purification plant for the removal of iron.

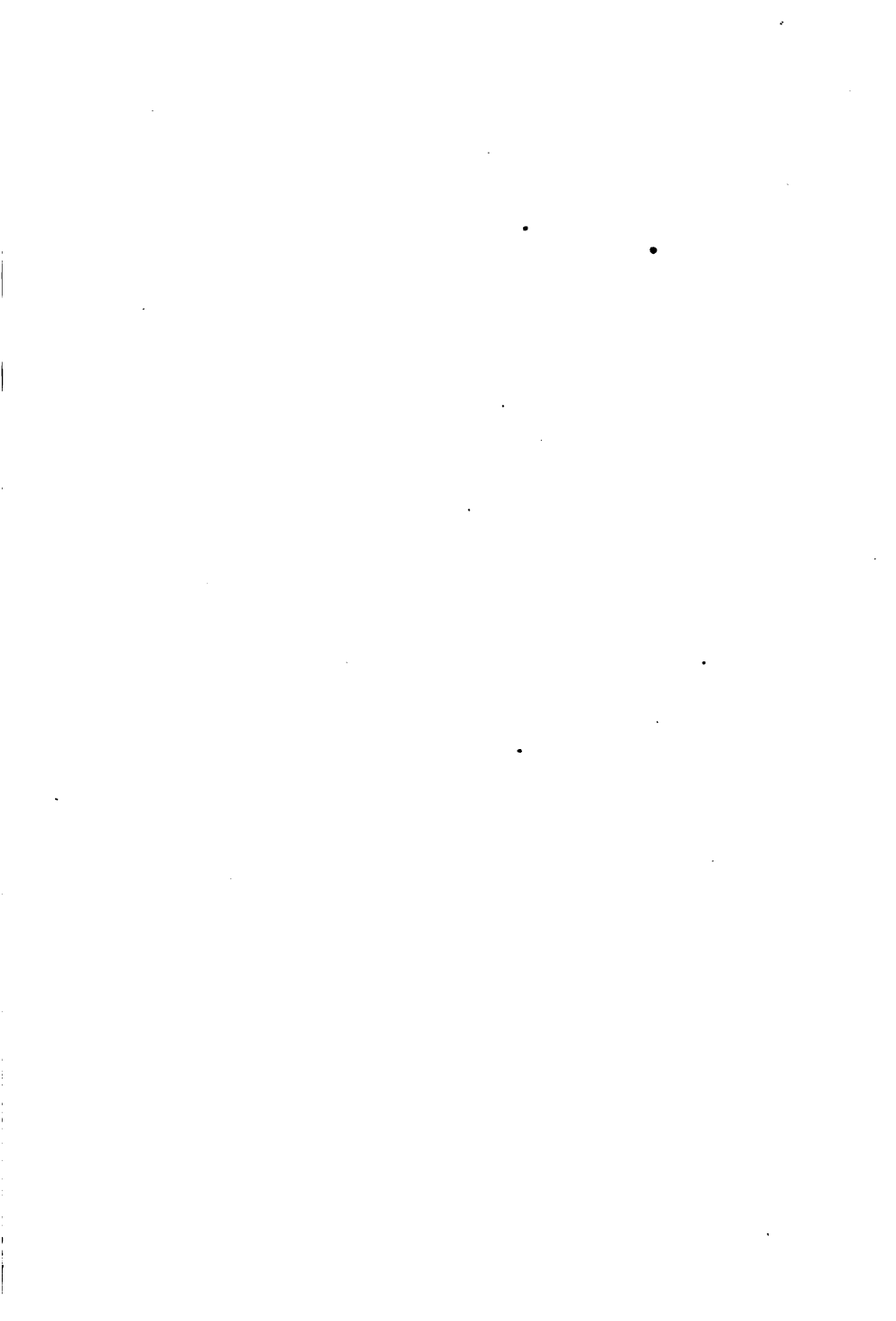
167. Describe a purification plant for softening water for municipal purposes.











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